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1a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED			1b. RESTRICTIVE MARKINGS NONE		
2a. SECURITY CLASSIFICATION AUTHORITY			3. DISTRIBUTION / AVAILABILITY OF REPORT APPROVED FOR PUBLIC RELEASE; DISTRIBUTION UNLIMITED.		
2b. DECLASSIFICATION / DOWNGRADING SCHEDULE					
4. PERFORMING ORGANIZATION REPORT NUMBER(S)			5. MONITORING ORGANIZATION REPORT NUMBER(S) AFIT/CI/CIA- 89-001		
6a. NAME OF PERFORMING ORGANIZATION AFIT STUDENT AT Univ of Texas - San Antonio		6b. OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION AFIT/CIA		
6c. ADDRESS (City, State, and ZIP Code)			7b. ADDRESS (City, State, and ZIP Code) Wright-Patterson AFB OH 45433-6583		
8a. NAME OF FUNDING / SPONSORING ORGANIZATION		8b. OFFICE SYMBOL (If applicable)	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER		
8c. ADDRESS (City, State, and ZIP Code)			10. SOURCE OF FUNDING NUMBERS		
			PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.
			WORK UNIT ACCESSION NO.		
11. TITLE (Include Security Classification) (UNCLASSIFIED) Comparison of Microleakage of Composite Resin Veneering Systems at the Alloy Interface					
12. PERSONAL AUTHOR(S) Luis James Blanco					
13a. TYPE OF REPORT THESIS/DISSERTATION		13b. TIME COVERED FROM TO	14. DATE OF REPORT (Year, Month, Day) 1988		15. PAGE COUNT 84
16. SUPPLEMENTARY NOTATION APPROVED FOR PUBLIC RELEASE IAW AFR 190-1 ERNEST A. HAYGOOD, 1st Lt, USAF Executive Officer, Civilian Institution Programs					
17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)		
FIELD	GROUP	SUB-GROUP			
19. ABSTRACT (Continue on reverse if necessary and identify by block number)					
DTIC ELECTE FEB 13 1990 S D D					
90 02 12 026					
20. DISTRIBUTION / AVAILABILITY OF ABSTRACT <input checked="" type="checkbox"/> UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT. <input type="checkbox"/> DTIC USERS			21. ABSTRACT SECURITY CLASSIFICATION UNCLASSIFIED		
22a. NAME OF RESPONSIBLE INDIVIDUAL ERNEST A. HAYGOOD, 1st Lt, USAF			22b. TELEPHONE (Include Area Code) (513) 255-2259	22c. OFFICE SYMBOL AFIT/CI	

COMPARISON OF MICROLEAKAGE OF COMPOSITE RESIN VENEERING  
SYSTEMS AT THE ALLOY INTERFACE

A

THESIS

Presented to the Faculty of

The University of Texas Graduate School of Biomedical Sciences

at San Antonio

in Partial Fulfillment

of the requirements

for the Degree of

MASTER OF SCIENCE

by

Luis James Blanco, B.S., D.M.D.

San Antonio, Texas

September, 1988

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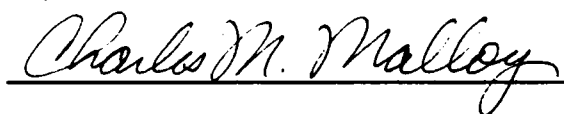
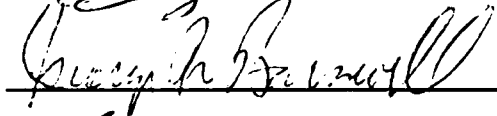
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Luis James Blanco

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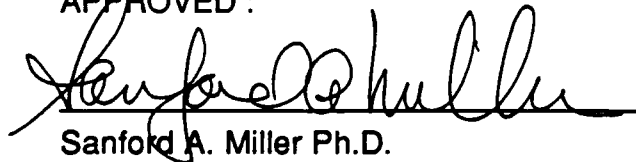
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## **DEDICATION**

I would like to dedicate this document to the best wife in the world, Adita and to my dear children, Jose, P.J. and Barbara. It was with their patience, understanding and support that I was able to achieve this goal.

## **ACKNOWLEDGEMENTS**

I wish to thank Dr. Barry K. Norling (Associate Professor and Head of the Division of Biomaterials) for his valuable time and sharing of his knowledge, and use of his laboratory. He was the driving force behind this research project and a constant source of encouragement.

Dr. David L. Carnes Jr. (Assistant Professor for the Department of Endodontics) for sharing his experience in the area of radioisotope handling and for opening his laboratory to me.

Dr. Charles M. Malloy (Resident Training Officer, Wilford Hall Medical Center) who has taught me Prosthodontics and has been a source of encouragement.

Special thanks to Dr. George M. Barnwell (Assistant Professor for the Department of Pathology) for his suggestions and assistance in design of the statistical analysis.

Dr. E. Steven Duke (Associate Professor for the Department of Restorative Dentistry) for his suggestions in methods and materials and planning of this project. Dr. Garrett E. Hartman (Assistant Chairman for the Department of Prosthodontics, Wilford Hall Medical Center) for serving on my graduate committee.

Mr. Ruben G. Urrutia and Mr. Manuel Angel for their assistance in specimen preparation.

Mr. Robert (Mac) McConnel, C.D.T. for helping me with the instrumentation on this research and Mr. Robert Wood for assistance in the statistical analysis.

A special thanks to Mrs. Ann Blanco for her editorial contributions.

# COMPARISON OF MICROLEAKAGE OF COMPOSITE RESIN VENEERING SYSTEMS AT THE ALLOY INTERFACE

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The lack of adhesion of acrylic resin to the metal of the resin veneer restoration required methods of mechanical retention. Poor color stability due to percolation of oral fluids at the metal-resin interface and breakdown of the acrylic resin were factors that have limited the acceptance and widespread use of this restoration. These were due to a high differential in coefficients of thermal expansion between the resin and metal, high water sorption, and high polymerization shrinkage,

Several new methods of retention have been developed to improve adhesion of the composite resin to metal. In addition, improvements made in composite resins make the composite-resin veneer restoration a viable solution when the metal-ceramic restoration is contraindicated. (ms) ←

The purpose of this investigation was to measure the amount of microleakage at the resin-alloy interface of three composite resin veneering materials and three new retention systems. The control specimens relied solely on microbeads for retention.

One hundred and twenty type IV gold alloy specimens, one centimeter square, and 0.5 millimeters thick, were distributed in a 4 X 3 arrangement. There were twelve groups of ten specimens each. Four methods of retention were used: Silicoater (Kulzer Co.), Lee metal primer (Lee Pharm.), 4-META (Parkell) and microbeads (ESPE-Premier). These retention systems were used with three composite resins:

Visio-gem (ESPE-Premier), Dentacolor (Kulzer Co.) and Elcebond (Teledyne-Hanau). The composite resins were processed in cylindrical specimens, according to manufacturers' instructions, at the center of the gold alloy castings (4.85mm diameter).

These simulated restorations were thermocycled at 6-60°C for 2,136 cycles at 30 sec. dwell, immersed in a solution of 0.2 mCi of  $\text{Ca}^{45}$  for two hours and sectioned with an Isomet diamond saw. The specimens were placed face down for five days on dental x-ray film for autoradiography. The resin-metal interfacial exposure on the dental film was measured with a Gaertner traveling microscope.

All of the 4-META specimens separated during thermocycling. Lee specimens survived the thermocycling procedure. However, more than 60% of the specimens separated during sectioning with the Isomet saw, rendering a population too small for statistical analysis. A two-way ANOVA demonstrated a significant statistical difference between Silicoater and microbead specimens ( $p < 0.001$ ). A subsequent Tukey's Multiple Comparison test ranked microleakage: Silicoater/Visio-Gem less than Beads/Visio-Gem, Silicoater/Elcebond less than Beads/Elcebond, Silicoater/Dentacolor less than Beads/Dentacolor ( $p < 0.001$ ). The type of composite resin was found to be significant ( $p = 0.021$ ) when used with the Silicoater adhesive system. After a Tukey's Multiple Comparison Test ( $P = 0.025$ ), the Silicoater/Elcebond combination exhibited higher microleakage values when compared to Silicoater/Visiogem and Silicoater/Dentacolor. However, mean values of microleakage of Silicoater/Elcebond are still very low when compared to microbeads.

Based on the results of this study it can be stated that the Silicoater was the most effective chemical retention system to resist microleakage when compared to a mechanical retention method.

## **TABLE OF CONTENTS**

	Page
Title .....	i
Approval .....	ii
Dedication .....	iii
Acknowledgements .....	iv
Abstract .....	v
Table of Contents .....	vii
List of Tables .....	ix
List of Figures .....	x
List of Plates .....	xi
I. INTRODUCTION .....	1
II. LITERATURE REVIEW .....	3
A. The Acrylic Resin Veneer Restoration .....	3
B. The Metal - Ceramic Restoration .....	4
C. Composite Resins .....	5
D. Composite Resin Veneering Systems .....	8
E. Base Metal Alloys .....	9
F. New Methods of Retention .....	9
G. Microleakage Studies .....	11
H. Autoradiography .....	11
I. Summary .....	12
J. Problem Statement .....	13
III. RESEARCH OBJECTIVES .....	14
IV. METHODS AND MATERIALS .....	15
A. Experimental Plan .....	15
B. Preliminary Preparations .....	15
C. Placement of Adhesive and Composite Resin .....	21
D. Thermocycling of Specimens .....	27
E. Radioisotope Procedure .....	27
F. Sectioning Procedure .....	31
G. Autoradiography .....	31
H. Measuring Procedure .....	31



I.	Statistical Analysis.....	36
V.	RESULTS .....	37
VI.	DISCUSSION .....	45
VII.	SUMMARY .....	56
	Appendix .....	57
	Literature Cited .....	67
	Vita .....	72

## **LIST OF TABLES**

	Page
Table 1. Specimen Arrangement .....	16
Table 2. Means and Standard Deviations of Microleakage of Retention and Resins.....	38
Table 3. Means and Standard Deviations of Microleakage of Retentive Systems .....	39
Table 4. Summary Table for Two-Way Analysis of Variance of Microleakage and Types of Retention.....	41
Table 5. Summary Table .....	42
Table 6. Tukey's Multiple Comparision Test Table .....	44

## **LIST OF FIGURES**

	Page
Figure 1. Cross-section View of Specimen Preparation .....	25
Figure 2. Specimen Placement Over Radiographic Film .....	34
Figure 3. Cell Means Graph .....	40
Figure 4. Molecular formula of 4-META .....	47
Figure 5. Mechanism of adhesion of 4-META .....	48
Figure 6. Mechanism of adhesion of Silicoater .....	52

## LIST OF PLATES

	Page
Plate 1. Plastic Bead Specimens Sprued .....	18
Plate 2. Cast Specimen Demonstrating Bead Arrangement .....	19
Plate 3. Cast Bead Specimens .....	20
Plate 4. Silicoater Specimens .....	22
Plate 5. Gasket on Silicoated Specimen .....	23
Plate 6. Punch Apparatus .....	24
Plate 7. Composite Resin Processing .....	26
Plate 8. Representative Specimens after Polymerization .....	28
Plate 9. Representative of Completed Specimen without any Composite Resin Flash .....	29
Plate 10. Thermocycling Apparatus .....	30
Plate 11. Placement of Specimen in Radioisotope Solution .....	32
Plate 12. Isomet Diamond Saw Sectioning Specimen .....	33
Plate 13. Placement of Sectioned Specimen over Radiographic Film .....	35
Plate 14. Representative of Lee Metal Primer after Thermocycling .....	50
Plate 15. Autoradiographs .....	54

## **I. INTRODUCTION**

The resin veneered restoration was an esthetic alternative in fixed partial dentures prior to the advent of the metal-ceramic crowns. The desirable features were ease of fabrication, good color matching , repair without removal from the oral cavity, and allowance of a more conservative tooth preparation than the metal-ceramic. The main disadvantages were reliance on beads, loops and peripheral undercuts as means of mechanical retention due to the lack of adhesion of acrylic resins to gold alloys, poor color stability due to percolation of oral fluids at the resin-metal interface, and low resistance to toothbrush abrasion. If chemical means could be used to achieve resin-metal bonding, 1) esthetics could be improved because of a more uniform layer of the opaque and composite resin, and 2) percolation of fluids at the metal-opaque interface could be reduced or eliminated.

Metal-ceramic restorations have replaced the acrylic resin restoration because of their esthetics and durable qualities. However, accidental fracture due to failure of the porcelain-metal bond has been a problem. Repair of porcelain utilizing direct-repair systems is regarded as interim treatment. Another problem with porcelain is its high abrasiveness when opposed by natural dentition.

Composite resins replaced the acrylic resins and have been continuously improved over the years. They are more dimensionally stable because of lower thermal coefficient of expansion, lower water sorption, lower dimensional changes on setting and greater resistance to toothbrush abrasion. Recent improvements in composite resins include the use of visible light polymerization. This permits incremental placement of the material permitting a more homogeneous polymerization with less shrinkage.

Several new composite resin veneering systems are now available, including Visio-Gem (ESPE-Premier), Dentacolor (Kulzer Co.) and Elcebond (Teledyne-Hanau). These visible light polymerizing composite resin veneering systems are more resistant to wear and more color stable than previous acrylic resin systems.

Microbeads, loops and undercuts in the metal substructure have been the most widely used means to mechanically retain the acrylic and composite resin to noble metals. Three new methods of retention are now available: Silicoater (Kulzer Co.), Lee metal primer (Lee Pharm.) and 4-META (Parkell).

This investigation evaluates microleakage of three new methods of retention and microbeads in combination with three composite resins, utilizing  $\text{Ca}^{45}$  as a radioisotope marker.

## **II. LITERATURE REVIEW**

### **A. The Acrylic Resin Veneer Restoration**

The acrylic resin veneer crown and fixed partial denture was the most esthetic and durable alternative prior to the development of the metal-ceramic crown. The acrylic veneer resins were sufficiently translucent to permit excellent shade matching due to the blending of colors from adjacent teeth (Johnson et al., 1971; Phillips 1982). The resins were adequately opaque to mask the dark metal with a minimum thickness of 0.6 mm (Long, 1968) to 1.0 mm (Johnson et al., 1971). This contributed to a more conservative tooth preparation than that needed for the metal-ceramic restoration (Long, 1968; Tylman, 1970). In addition, the veneering material could be repaired intraorally without the need to remake the entire prosthesis or restoration (Tylman, 1970).

However, many disadvantages outweighed the few benefits. The lack of adhesion of the acrylic resin (polymethylmethacrylate) to the casting made it necessary to rely on beads, loops and peripheral reverse bevels for mechanical retention (Dykema et al., 1958; Johnson et al., 1971; Long, 1968). Other deficiencies included a high differential in coefficients of thermal expansion between the resin and metal, high water sorption, and high polymerization shrinkage (Tylman, 1970; Johnson et al., 1971; Phillips, 1982). Low values for proportional limit and modulus of elasticity of the resin required a strong metal substructure for support and protection from occlusal loads to prevent plastic deformation of the veneering materials (Peyton and Craig 1963; Phillips 1982).

Poor color stability of the acrylic resin was attributed partially to the leakage of oral fluids at the resin-metal interface (Lamstein and Bleckman, 1956; Swartz et al., 1957; Peyton and Craig 1963; Issa, 1968; Phillips, 1982). Gold plating the non noble metal substructure prevented tarnishing of the casting (Dykema et al.,

1958; Johnson et al., 1971).

The low resistance to toothbrush abrasion of resin veneers caused clinicians to recommend soft toothbrushes and toothpastes with no coarse abrasives (Dykema, 1958; Johnson et al.; 1971; Phillips, 1982) and proper brushing techniques (Phillips, 1982).

The original acrylic resins employed for crown and fixed partial denture applications were acrylic, vinyl-acrylic polyester and epoxy-resin formulations (Peyton and Craig, 1983). The acrylic and vinyl-acrylic materials were supplied as powder-liquid systems and required flasking like denture base resins. Another resin, a poly (methyl methacrylate) and a methacrylate ester with low volatility, produced a gel which could be polymerized in a pre-heated oven at 135°C without the need of flasking. This facilitated the procedure and prevented warping of the framework (Craig, 1985).

## **B. The Metal-Ceramic Restoration**

The metal-ceramic restoration has been one of the treatments of choice in situations where an esthetic complete coverage is required for a single tooth (Phillips, 1982). *The esthetic qualities of porcelain remain a main advantage.* The ability to reproduce, in great detail, the characteristics and peculiarities of an individual tooth within a given dentition has been reported (Tylman and Malone, 1978).

Unfortunately, some disadvantages have been reported. Accidental fracture of the porcelain or failure of the bond to the metal substructure have been common (Highton et al., 1979). Direct repairs can be performed with composite filling resins. However, they are regarded as interim treatments because of lack of adhesion, poor color stability, high coefficient of thermal expansion and poor abrasion resistance of the repair materials (Phillips, 1982). Organo-silane



coupling agents used in combination with acrylic and composite resin have been evaluated for porcelain repair (Eames et al., 1977; Eames and Rogers, 1979; Highton et al., 1979; Nowlin et al., 1981). Resulting bond strengths of repaired porcelain were as high as 48% (Highton et al., 1979) and as low as 18% (Nowlin et al., 1981). Further research has been suggested to develop a metal primer with adequate adhesive properties to repair porcelain, because those used today have low bond strengths (Tjan et al 1987).

The surface roughness characteristics and the high hardness value of dental porcelain and the resulting abrasion to opposing natural teeth or other restorative materials has been reported. Enamel surfaces were found to wear at an accelerated rate when occluding against porcelain as compared to enamel occluding against gold (Monasky and Taylor, 1971).

### **C. Composite Resins**

Since the introduction of composite resins to the dental profession, there has been an improvement in the quality of the direct filled anterior restorations. Compared to its predecessor (the unfilled acrylic resins) the composite resins have a lower thermal coefficient of expansion, lower water sorption, smaller dimensional change on polymerizing, and higher resistance to abrasion (Craig, 1985; Leinfelder, 1985).

Composite resins are three component systems which include an organic phase (resin matrix), an interfacial phase (silane coupling agent), and a dispersed phase (inorganic filler).

The organic phase components include 2,2-bis[4(2-hydroxy-3methacryloyloxy-propyloxy)-phenyl]propane (BIS-GMA), modified BIS-GMA, urethane diacrylates, triethyleneglycol dimethacrylate (TEG-DMA) and a number of other reactive diluents (Bowen, 1962; Lutz et al.,

1983; Craig, 1985). The interfacial phase consists of bipolar molecules, mainly organosilanes, which are used as coupling agents to bond the inorganic fillers and the organic matrix (Lutz et al., 1983; McCabe, 1984). The dispersed phase (inorganic fillers) may consist of quartz, boro-silicate glass, lithium-aluminum-silicate glass, or barium-glass (Craig, 1985).

One method of classifying composite resins has been by the particle size of the inorganic filler. In the conventional composite resins the inorganic particles are irregular in shape and the particle size ranges from 15 to 35 $\mu$ m. For an intermediate composite resin, the range in particle sizes may be from 1 to 5  $\mu$ m and for the microfilled composite (pyrolytic or precipitated particle of silica) particles are generally  $\leq .04\mu$ m (Leinfelder, 1985).

The conventional composite resins were an improvement over the unfilled resins. Unfortunately, after a period of time the surface would appear rough due to poor abrasion resistance of matrix resin in comparison with filler. The introduction of the microfilled class of composite resins, without the much larger quartz and glass fillers, improved the surface smoothness (Phillips, 1982; Craig, 1985).

The microfilled composite resins are less highly filled than the conventional and intermediate composite resins. The greater surface area of the small silica particles requires a greater volume of matrix material resulting in filler contents of only 30 to 50% (Phillips, 1982; Craig, 1985). This decrease in inorganic filler in the resin matrix is considered responsible for the greater water sorption and higher coefficient of thermal expansion than those with conventional or intermediate composite resins (Swartz et al., 1982). It has been reported that thermal cycling of the microfills will cause marginal leakage because of the increased difference in coefficient of thermal expansion between these microfilled resins and the metal substructure (Hembree, 1983). Color fading has

also been reported (St. Germain, 1985). Also, pitting of the surface has been noted, probably due to a failure in bonding of the prepolymerized particles and the resin matrix (Leifelder, 1985). When used as Class V restorations, microfills wear twice as fast as conventional composites (Leifelder, 1985).

Composite resins also may be classified by the method of polymerization. Some traditional composite resins are chemically initiated (autopolymerized) with the use of a benzoyl peroxide initiator and a tertiary amine accelerator. These materials are supplied as two components; two pastes or a powder and a liquid. Other composite resins may be photoinitiated with exposure to either ultraviolet light (365nm) or visible blue light (450nm). These are supplied as a single component (one paste) (Craig, 1985). With the UV light activated composite resins an organic compound (e.g. benzoin methyl ether) is substituted for the benzoyl peroxide (Craig, 1981).

For the visible light system a diketone, such as camphoroquinone, is used with an organic amine as the initiator (Craig, 1981; Phillips, 1982). Many advantages have been demonstrated with the light activated composites compared to the chemically autopolymerized resins. The light cured systems provide a higher degree of polymerization (Lutz et al., 1983). Being a one paste system, they do not require mixing, which results in less porosity. An incremental placement technique yields an improved margin adaptation (Lutz et al., 1983). Improved wear resistance of a light activated composite resin versus chemically activated has been reported (Rice et al, 1984). And, as a group, light activated composite resins allow a more optimal match in color, shape, and translucency, (Lutz et al., 1983; McCabe, 1985) and are more color stable than autopolymerized composite resins (Asmussen, 1985).

#### **D. Composite Resin Veneering Systems**

Recently, a new generation of composite resin veneering materials for prosthodontic applications has been introduced. These composite resins appear to have improved qualities over previous resin-based veneering materials. Preliminary reports of increased resistance to toothbrush abrasion with three new composite veneering materials compared to an acrylic resin were noted, however, tensile strengths were not as high as for acrylic resin (Nathanson et al., 1985).

All three of the composite resins evaluated in this microleakage study: Visio-Gem, Elcebond and Dentacolor, are polymerized by special visible light curing units.

Visio-Gem is described as "a hybrid microfilled composite with splintered prepolymerized particles and agglomerated microfiller complexes" (Strohaver and Mattie, 1987). It contains two distinct types of filler particles. One is a large irregular shaped pre-polymerized particle of 200 $\mu$ m or more. The other phase contains agglomerations of microfiller particles. Visio-Gem has been described as a bifunctional methacrylate filled with pyrogenic silicon dioxide (Product information, ESPE-Premier). Of those tested in one study, Visio-Gem had the fewest voids of the light cured composite resins (Strohaver and Mattie, 1987).

Elcebond consists of a 20% dimethacrylate matrix, 26% organic filler and silane (coupling agent), and 54% pyrogenic silicic acid. It contains splintered prepolymerized particles with spherical beads that average 2 $\mu$ m in diameter. Elcebond has been classified as a hybrid microfilled composite with splintered prepolymerized and spherical particles (Strohaver and Mattie, 1987).

Dentacolor's matrix is described as a multifunctional methacrylic ester. The composite contains 72% pyrogenic silica by weight. It has been classified as "a heterogeneous microfilled composite with splintered prepolymerized particles"

(Strohaver and Mattie, 1987).

### **E. Base metal alloys**

Base metal alloys have been electrolytically and chemically etched to increase the mechanical retention considerably with acrylic (Tanaka et al., 1979) and composite resins (Bertolotti and Napolitano, 1983). However, the biocompatibility of base metal alloys containing nickel and beryllium has been questioned recently (Blanco-Dalmau, 1982; Blanco-Dalmau et al., 1984; Covington et al., 1985). Gold-base alloys are still considered the most biocompatible alternative, but cannot be electrolytically or chemically etched like base metal alloys.

### **F. New Methods of Retention**

New methods of adhesion and retention to metal have been introduced in the past years. The OVS system (De Trey Dentsply, FR Germany) features tin plating of base metal and gold alloy. This european system has been described by Veen, Bronsdijk and Poel (1985). The Kura-Ace (Kuraray, Japan) is another similar system used in Japan (Yamashita et al., 1986). Tin plating is applied by an electrochemical process and then the tin is oxidized to produce a surface suitable for chemical bonding of composite resin materials.

Silicoater, another new method of retention, relies on pyrogenic silica coating of the metal surface. This produces a chemical bond between the metal and the composite resin veneering material. A silicon-oxide-carbon ( $\text{SiO}_x\text{C}$ ) coating is formed ( $0.5\mu$ ), to which a silane coupling agent is applied ( $0.5\mu$ ), followed by the composite resin. The  $\text{SiO}_x\text{C}$  alone does not provide an adequate surface to bond with the composite resins. The surface -OH terminals of silicon dioxide ( $\text{SiO}_2$ ) do not react with the end groups of the composite resins.

However, a suitable substrate is produced by condensing surface -OH groups with the -OH groups of the hydrolyzed silane. Pendant polymerizable groups of the silane polymerize with methacrylate groups of the composite resin. This makes possible a chemical bond between the metal and the composite resin (Kulzer Product information).

Lee Metal Primer consists of an epoxy resin metal primer with an opaquer. It is applied and baked to the surface of the metal (at a thickness of 0.0005"), promoting adhesion and masking the metal. This metal primer was developed as an opaquer-adhesive promoter for the non acid-etched resin-bonded fixed partial denture (Maryland Bridge). Tests made by Lee's own laboratories indicate adhesive strengths higher than those produced by other composite resin luting agents to non-etched metal ( Lee Pharm. Product information, 1985). This material is also recommended as an opaquer-adhesion promoter with composite resin veneering systems.

A fourth method of retention is 4-Methacryloyloxyethyl trimellitate anhydride (4-META). This adhesive was developed by Matsuhara et al. in 1962. The combination of 4-META with tributyl borane oxide (TBB-O) (Takeyama et al 1978), produced a commercial product, Super-Bond C & B (Masuhara, 1982), which is adhesive to dental alloys and tooth surface. 4-META is an adhesive resin that has been reported to be effective as an opaquing media and adhesion promoter over base metals in acrylic veneered crowns (Tanaka et al., 1981, Tanaka et al.,1986). 4-META was developed as a bonding agent for the acid-etched resin-bonded fixed partial denture (Maryland Bridge). It is bound to the oxide layer created by etching Ni-Cr alloys with HCl, and later oxidizing with  $\text{HNO}_3$  (Tanaka et al., 1981; Xin-yi Yu et al., 1987). The usefulness with gold alloys has not been reported.

With the introduction of new systems of adhesion to metal and a new

generation of composite resin veneering materials for prosthodontic applications, there has been new interest in the composite resin veneer restoration as a metal-ceramic alternative.

### **G. Microleakage Studies**

Lamstein and Bleckman (1956) demonstrated through a microbiologic and a methylene blue dye technique that there is leakage around resin veneers in gold crowns. Microleakage studies have been used primarily for the evaluation of direct restorative composite resin materials. The use of dyes and stains such as methylene blue, basic fuchsin (Crim and Mattingly, 1981; Fuchs, Hirshfeld and Grajower, 1985) and silver nitrate (Wu and Cobb, 1981) for the evaluation of marginal leakage has been reported. However, evaluation of dye penetration at the composite resin-metal interface is difficult. The difference in optical densities between the composite resin and metal provide poor dye contrast when transilluminated and evaluated with a light microscope.

Measurement of volume changes that occur in microleakage has been reported also (Arends, Veen and Schuthof, 1984).

The use of radioisotopes, such as for the evaluation of marginal leakage in gold crowns was first reported by Swartz and Phillips in 1957, utilizing a technique reported by Crawford and Larson a year earlier for evaluating amalgam microleakage.

### **H. Autoradiography**

"Autoradiography is a photographic method for recording the spacial distribution of radioisotope-labeled substances within a specimen material." (Kodak information, 1986) Specimens are immersed in a radioisotope solution and placed directly over radiographic film. This technique is called direct

exposure.  $\text{Ca}^{45}$  is a radioisotope which is a beta particle emitter. A beta ( $\beta$ ) particle is a negative charged particle emitted from the nucleus of an atom, having a mass and charge equal in magnitude to that of an electron (Martin and Harbison, 1972). In the case of  $\text{Ca}^{45}$ , a single emulsion film can be used such as Kodak Ultra-speed Dental film. (Kodak information, 1986) If microleakage has occurred, the radioactive emissions that remain in the specimen release energy to the silver halide grains in the emulsion layer of the photographic film rendering an image on the radiograph (autoradiograph) which can be located and measured and used as a permanent record.

The concentration of  $\text{Ca}^{45}$  (0.1 mCi/ml) first used for microleakage studies is still the most commonly used today (Phillips, et al 1961; Hembree and Taylor, 1984; Welsh and Hembree, 1985; Crim, Swartz et al, 1985). Other authors (Staffanou, Hembree et al, 1985), have used lower concentrations of  $\text{Ca}^{45}$  (0.1  $\mu\text{Ci/ml}$ ).  $\text{Ca}^{45}$  has not been the only radioisotope employed.  $\text{Na}^{22}$  was used by Bergman (1961) to evaluate the permeability of acrylic facings in gold crowns. Also,  $\text{I}^{131}$  has been used (Galan et al., 1976) for the evaluation of marginal leakage in composite resin restorations.

## **I. Summary**

Several new methods of retention have been developed to improve adhesion of the composite resin to metal. In addition, improvements made in composite resins make the composite resin veneered restoration a possible solution when metal-ceramic is contraindicated.

The purpose of this investigation was to measure the amount of microleakage at the resin-alloy interface of three composite resin veneering materials and three new retention systems. The control specimens rely solely on microbeads for retention.



## **J. Problem Statement**

The lack of adhesion of the acrylic resin to the metal substructure in the acrylic resin veneered crown made it necessary to use beads, loops and peripheral reverse bevels for mechanical retention. Poor color stability due to leakage of oral fluids at the resin-metal interface because of high differential in coefficients of thermal expansion between the resin and metal, high water sorption, high polymerization shrinkage and low resistance to toothbrush abrasion have limited the acceptance and widespread use of this restoration. The importance of this investigation is that it will address one factor, microleakage, to evaluate if chemical retention materials actually bond to metal.

## **Research Hypothesis**

The specimens treated with chemical adhesives will exhibit less microleakage at the acrylic-metal interface than the specimens relying on pure mechanical retention (microbeads).

## **Null Hypothesis**

There will be no difference in microleakage between specimens treated with chemical adhesives or microbeads.

### **III. RESEARCH OBJECTIVES**

The objective of this investigation is to evaluate the bond between veneering composite resin and metal substructure. Measurement of microleakage at the composite resin-alloy interface is used for this purpose. Three composite veneering materials studied in combination with three new retention systems were compared to microbeads, the most common retention system used at this time.

## **IV. METHODS AND MATERIALS**

### **A. Experimental Plan**

One hundred and twenty gold alloy specimens were distributed in a 4 X 3 arrangement, (Table 1) in twelve groups of ten specimens each. Four methods of retention were used: Silicoater (Kulzer Co.), 4-META (Parkell), Lee metal primer (Lee Pharm.) and microbeads (ESPE-Premier). These retention systems were used with three composite resin systems: Visio-Gem (ESPE-Premier), Dentacolor (Kulzer Co.) and Elcebond (Teledyne-Hanau). The composite resins were processed in cylindrical specimens, according to manufacturers' recommendations, to the center of the gold alloy casting. These specimens were thermocycled, immersed in a solution of  $\text{Ca}^{45}$  and later sectioned with an Isomet diamond saw. The specimens were placed face down on dental X-ray film for autoradiography. The resin-metal interfacial isotope penetration on the film was measured with a Gaertner traveling microscope.

### **B. Preliminary preparations**

One hundred and twenty plastic patterns were made by scoring 10mm square grids of 1.5 mm thick polycarbonate sheet (Plastic Supply Co., San Antonio, TX). The patterns were broken from the sheet and deburred by wet grinding on a 400 grit silicon carbide paper. Thirty patterns were masked by placing a teflon tape of 0.28mm thickness (0.011") with a 4.80 mm diameter hole over the center portion of the pattern. A thin layer of Visio-Gem light polymerized adhesive was applied to the surface. Sixteen retention microbeads (Visio-Gem 0.4mm) were applied to the painted surface utilizing a 40X power stereomicroscope (H & R Optical Systems, Boulder CO). The microbeads were individually placed to create a 4 by 4 bead square pattern with a diagonal

**Table 1. Specimen Arrangement**

RETENTIVE SYSTEMS	COMPOSITE RESINS		
	VISIO-GEM	ELCEBOND	DENTACOLOR
BEADS	1-10	11-20	21-30
SILICOATER	31-40	41-50	51-60
LEE	61-70	71-80	81-90
4-META	91-100	101-110	111-120

dimension of approximately 4 millimeters. The micro beads were carried to the pattern by means of the tip of red pencil moistened with Visio-Gem light polymerized adhesive.

Ten patterns were attached to a 3.25mm (1/8") diameter by 5.08cm (2") long polymethylmethacrylate rod runner bar with sticky wax utilizing an electric waxer (Almore Mini Waxer, Darby Dental Supply, NY) (Plate 1). A casting ring of 6.35cm (2.5") by 4.76mm (1 7/8") was used. The sprued patterns were later sprayed with Wax Pattern Cleaner (Jelenko, Armonk, NY) and allowed to dry.

The patterns were invested in vacuum mixed gypsum-bonded investment (Beauty Cast, Whip Mix Corp., Louisville, KY). The water to powder ratio was 14.5cc to 50 grams. The rings were lined with a single layer of wet asbestos substitute (Whip Mix Corp., Louisville, KY). After the ring was filled with the investment, it was placed in a 100°F water bath and allowed to set for 45 minutes (hygroscopic technique).

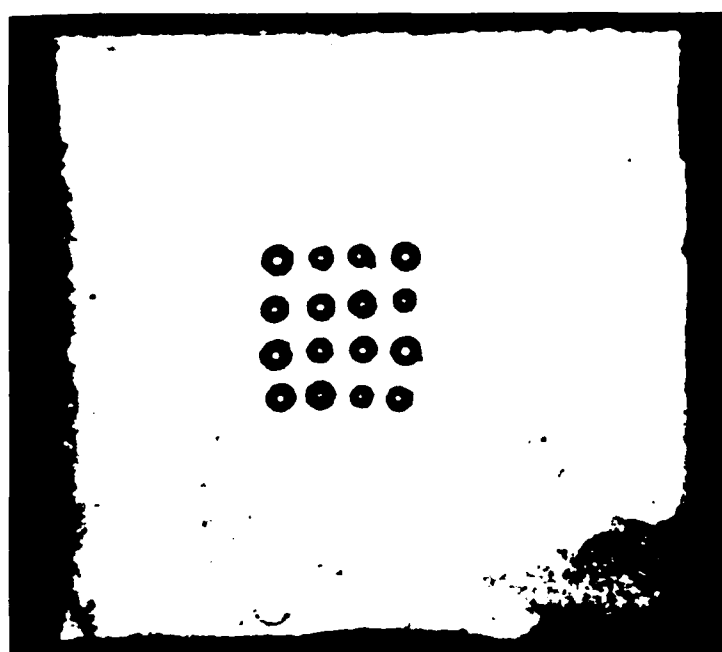
The rings were placed in a burnout oven at room temperature and thereafter heated to 900°F and sustained at this temperature for 1 hour. The patterns were cast in an ADA certified type IV noble alloy (Baker Inlay Extra Hard Type gold, Engelhard Inc., Carteret, NJ) (Plate 2). The alloy was melted with a natural gas-air torch and cast in a broken arm centrifugal casting machine. Rings were allowed to bench cool before removal of investment. Castings were air abraded with 50 micron aluminum oxide and the sprues removed with a thin 7/8" X .009" separating disk (Jelenko., Armonk, NY). Visual inspection of the castings ensured that the samples were devoid of nodules, distortion and rough surface. Those specimens with retentive beads were inspected for uniformity of placement (Plate 3). All specimens were cleaned with distilled water in a ultrasonic cleaner, rinsed with acetone and air dried with compressed air.

**Plate 1. Plastic Bead Specimens Sprued**

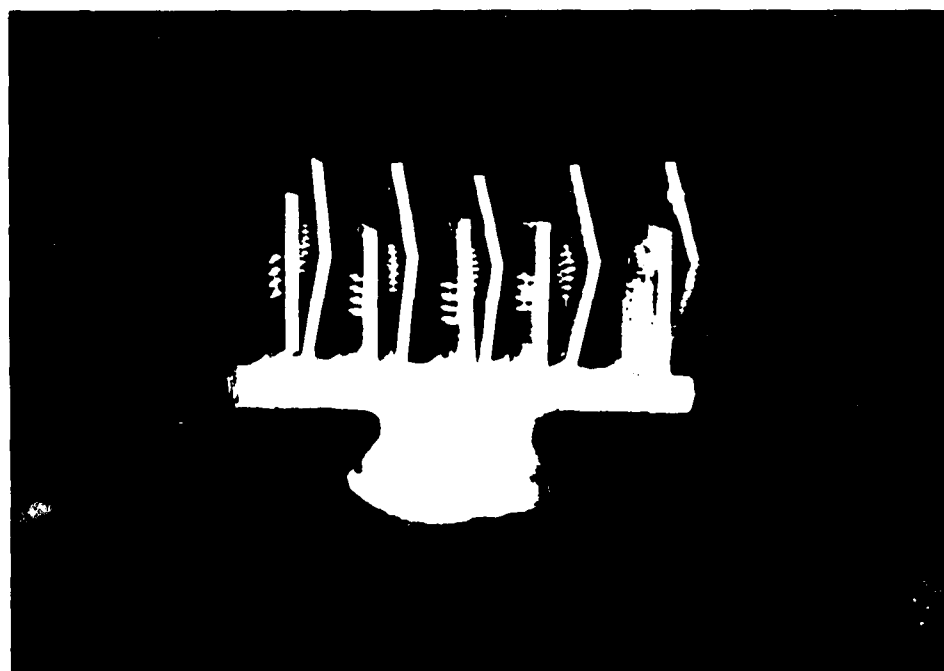


**Plate 2. Cast Specimen Demonstrating  
Bead Arrangement**





**Plate 3. Cast Bead Specimens**



### **C. Placement of adhesive and composite resin**

The following chemical adhesives were applied to the metal specimens utilizing manufacturers' instructions.

#### **Silicoater specimens**

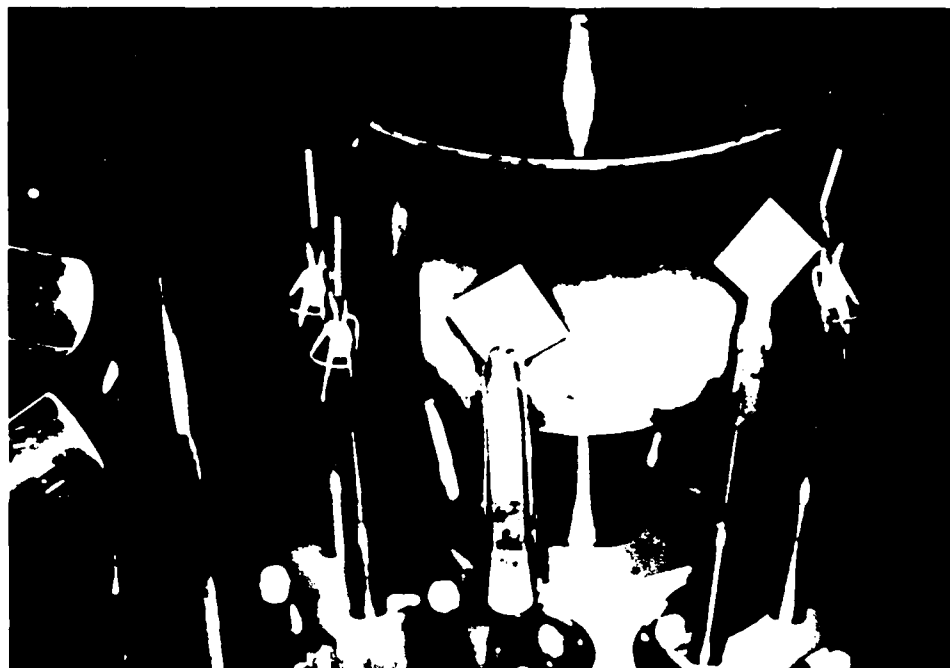
The Silicoater specimens were placed in a jar and cleaned with ethyl acetate (Siliclean) and left to air dry. The specimens were mounted on the Silicoater apparatus (Plate 4) where they were coated in the oxidizing region of a propane-tetraethoxysilane-oxygen flame for 5 minutes and left to cool for 3 minutes. After being cooled, the surface was painted with Silicoup (3-methacryloyloxypropyltrimethoxy-silane) adhesion primer solution and allowed to air-dry. To limit the area of composite resin application, a punched teflon tape with an inside diameter of 4.80mm was used as a gasket (Plate 5). A punch apparatus was designed and fabricated for this purpose (Plate 6). Composite resin was confined by means of a teflon tube with 6mm. outside diameter, 4.85mm. inside diameter and an overall height of 6mm. (Figure 1). The teflon tube was luted to the gasket by means of sticky wax placed with the electric waxer under a 10X power stereomicroscope (Plate 7). The teflon tape and tube was placed and composite resin was processed within one half-hour.

#### **Lee Metal Primer**

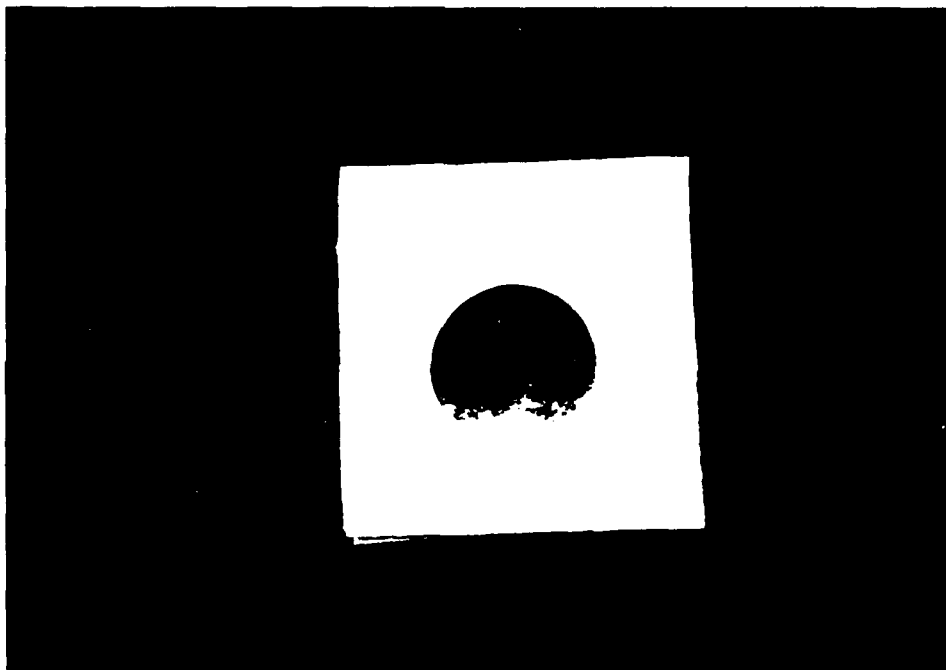
A thin coat of the primer was painted on the metal surface and allowed to dry for 15 minutes. The specimens were placed in a cold burnout oven (calibrated) and brought to 185°C (360°F) for ten minutes. The coated specimens were allowed to cool for 1 hour before placement of the teflon tape, tube and composite resin.

**Plate 4. Silicoater Specimens**

- A. Specimen Placement on Apparatus
- B. Specimen With SiO<sub>2</sub> C Layer

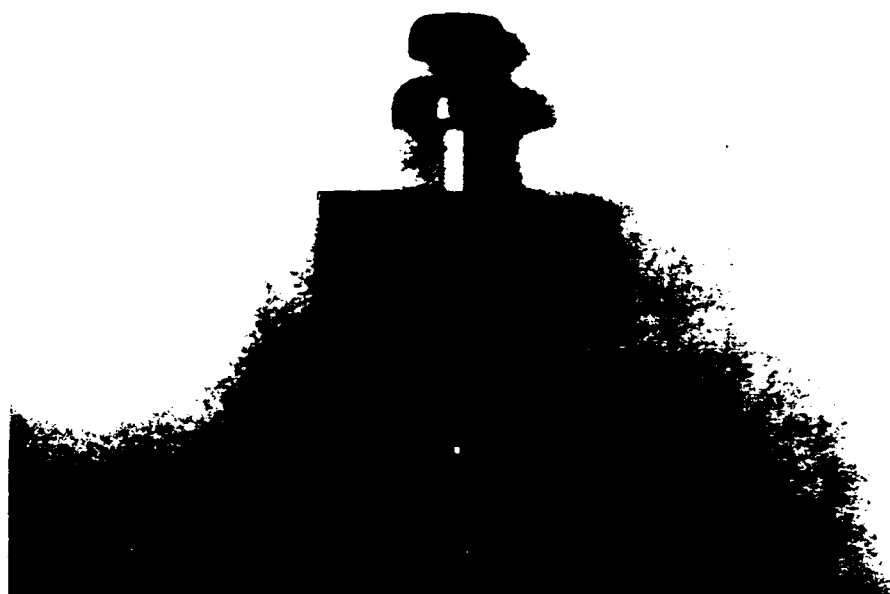


**Plate 5. Gasket on Silicoated Specimen**

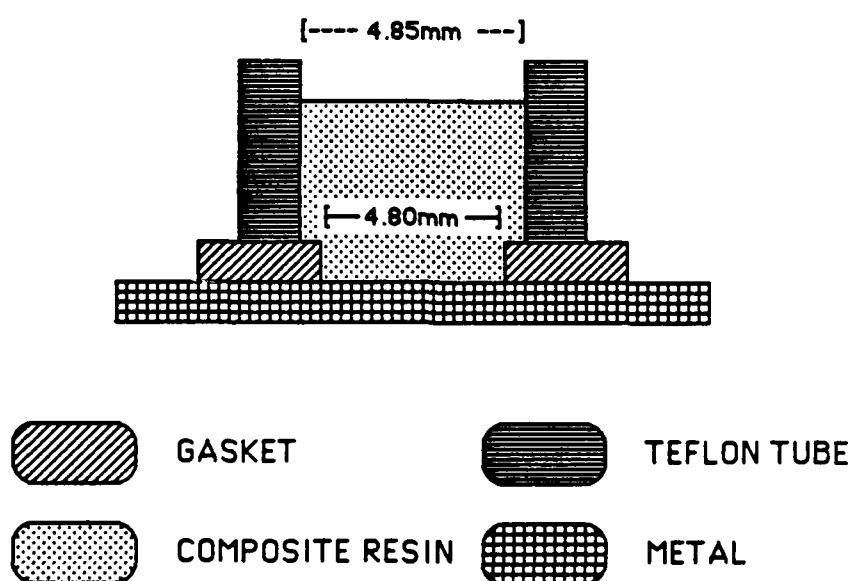




**Plate 6. Punch Apparatus**



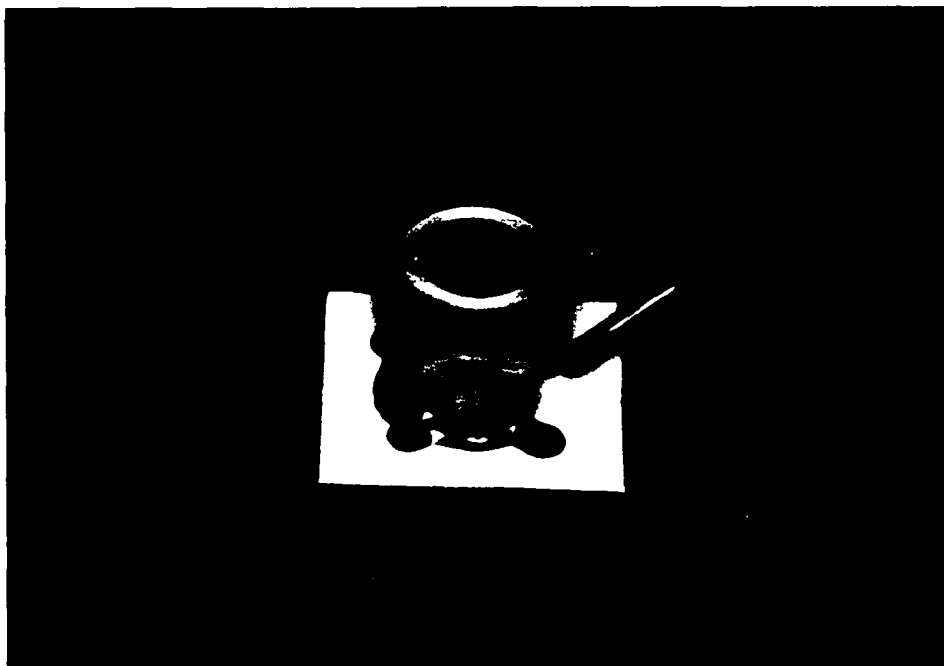
**Figure 1.** Cross-section view  
of specimen preparation



**Plate 7. Composite Resin Processing**

**A. Placement of Teflon Tube and Securement  
with Sticky Wax**

**B. Placement of Vision-Gem into Teflon Tube**



#### **4-META**

The 4-META liquid used was part of Parkell's Cover-Up kit. A thin coat was painted on the metal surface and allowed to dry 1 minute. The teflon gasket and tube was placed immediately after and the composite resin processed within 2 minutes.

Placement of the composite resins, dentin shade A-2 (Dentacolor, Visio-Gem and Elcebond) were made following the manufacturers' recommendations. A 2mm layer of composite resin was placed under a stereomicroscope (10X) in three increments and was not subjected to pressure (Plate 8). After the composite resin polymerized, the teflon tube was removed and the specimen inspected for composite resin flash (Plate 9). The polymerized composite resin was covered with clear nail polish up to 0.5mm. from interface to prevent absorption of the radioisotope into the composite resin. The specimens were placed in a 37°C distilled water bath for 24hrs.

#### **D. Thermocycling of specimens**

Specimens were thermocycled for 24 hours (2,136 cycles) in distilled water baths at 30 sec intervals (dwell time). (Crim, Swartz and Phillips, 1985). The thermocycling system consisted of two baths; one set at 6°C and the other at 60°C. Temperature was calibrated and maintained at  $\pm 2^\circ\text{C}$  in each of the baths by means of a thermostat (Plate 10).

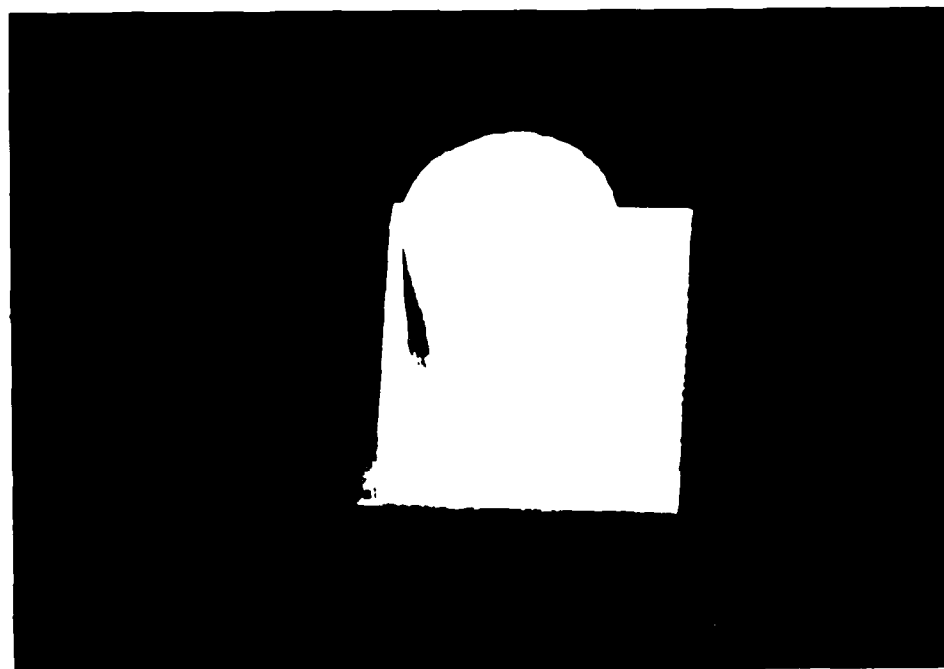
#### **E. Radioisotope procedure**

After drying the excess water from the specimens, they were placed in  $\text{Ca}^{45}$  isotope solution for 24 hours. Each specimen was placed individually in a 16mm diameter polystyrene well (Corning Cell Wells # 25820), in a 600 $\mu\text{l}$  radioisotope

**Plate 8. Representative Specimens after Polymerization**

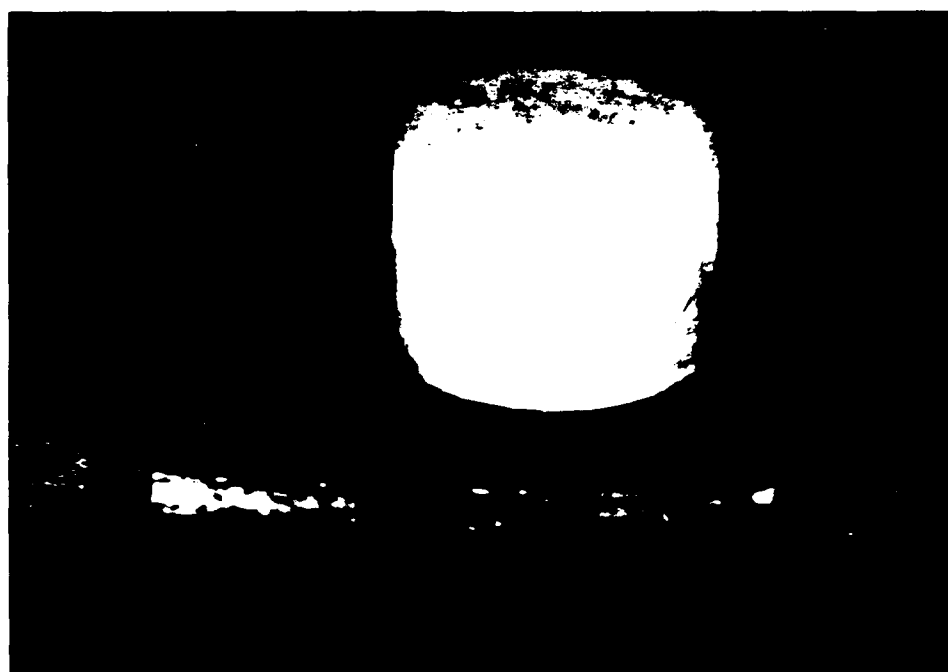
A. Top View

B. Lateral View





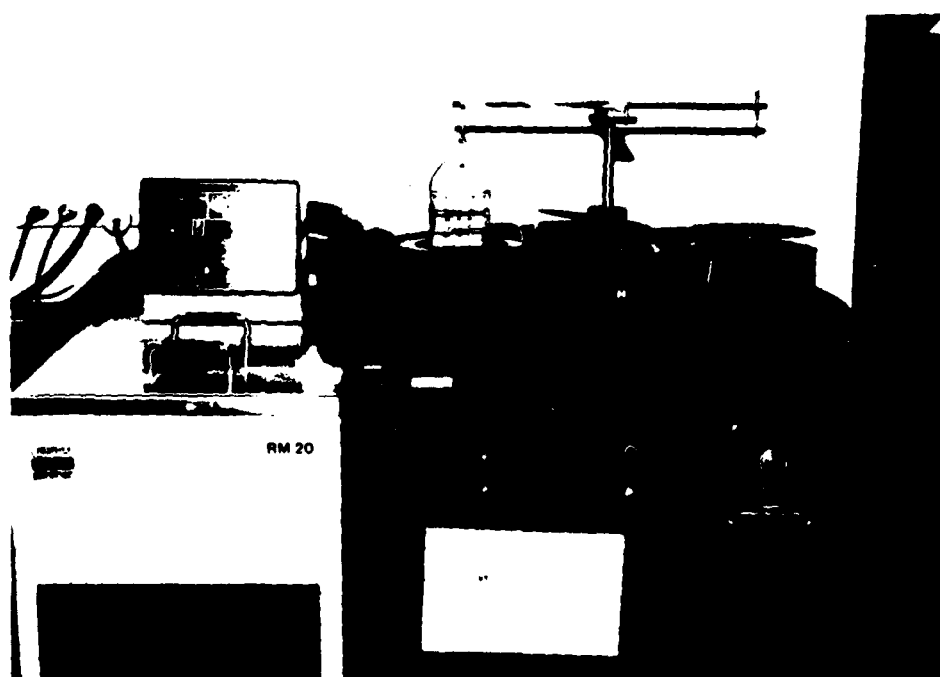
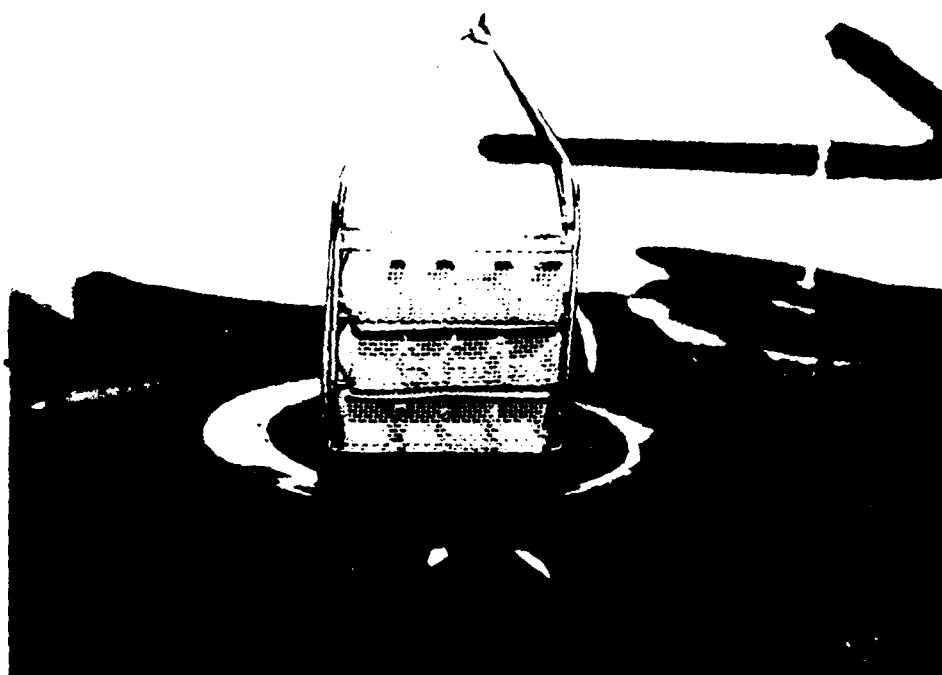
**Plate 9.** Representative of Completed Specimen without  
any Composite Resin Flash



**Plate 10. Thermocycling Apparatus**

A. Specimen Basket

B. Thermocycling Apparatus with Temperature  
Controlling Devices



solution and kept at 100% humidity (Plate 11). The concentration of the isotope was 0.2 millicuries per milliliter (mCi/ml). Verification of the concentration was made by diluting this solution 1/10 and counting duplicate 6 $\mu$ l aliquots in Liquid Scintillation solution and placing them through a Liquid Scintillation Spectrometer (Beckman model 1800). The specimens were carefully rinsed with distilled water for ten seconds to remove the excess isotope.

#### **F. Sectioning procedure**

The specimens were sectioned with a slow speed Isomet instrument (Buehler Ltd., Lake Bluff, IL). A diamond disk (4" X 0.012") lubricated with mineral oil was used to section the specimens in half, parallel to the cylinder axis. This exposed the composite resin-metal interface to be evaluated (Plate 12).

#### **G. Autoradiography procedure**

After sectioning, the samples were placed, sectioned side face down, directly on the emulsion side of Kodak Ultra-Speed Dental radiographic film (Eastman Kodak Co., Rochester, N.Y.) for 5 days (Figure 2). The specimens were stabilized against the film with a polymethylmethacrylate plate and rubber bands (Plate 13). The films were developed for 5.5 minutes in an automatic processor.

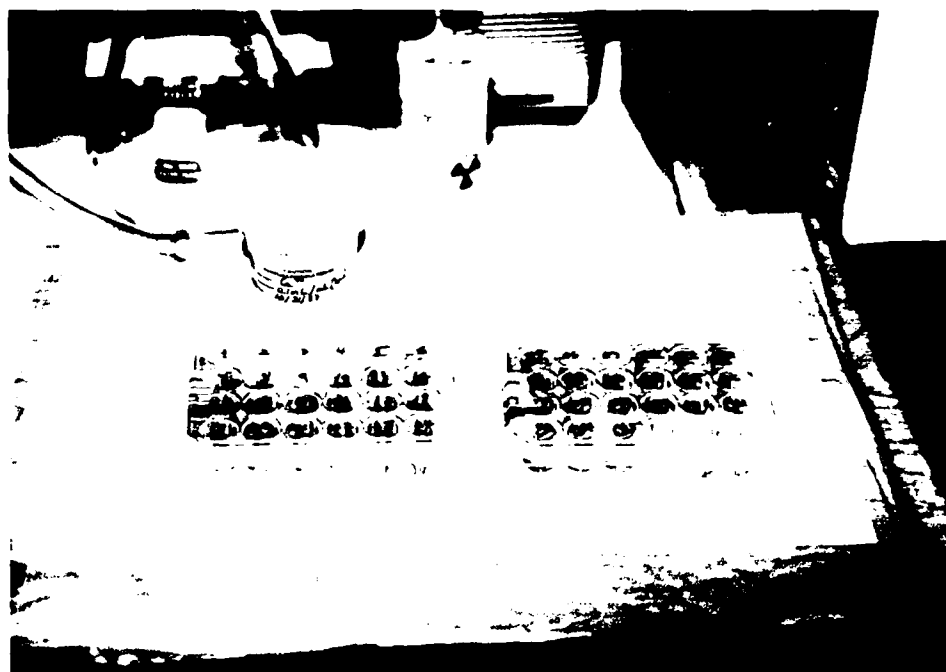
#### **H. Measuring procedure**

Radioisotope penetration depth of the autoradiographs was evaluated and measured with a Gaertner traveling microscope at 30X power to the nearest micron ( $\mu$ m). The composite resin diameter was measured first, and the radioisotope penetration thereafter. The cross-hairs of the eyepiece were aligned to the internal aspect of the notch created by the teflon gasket. Measurements were made from one notch to the other through the diameter of

**Plate 11. Placement of Specimens in Radioisotope Solution**

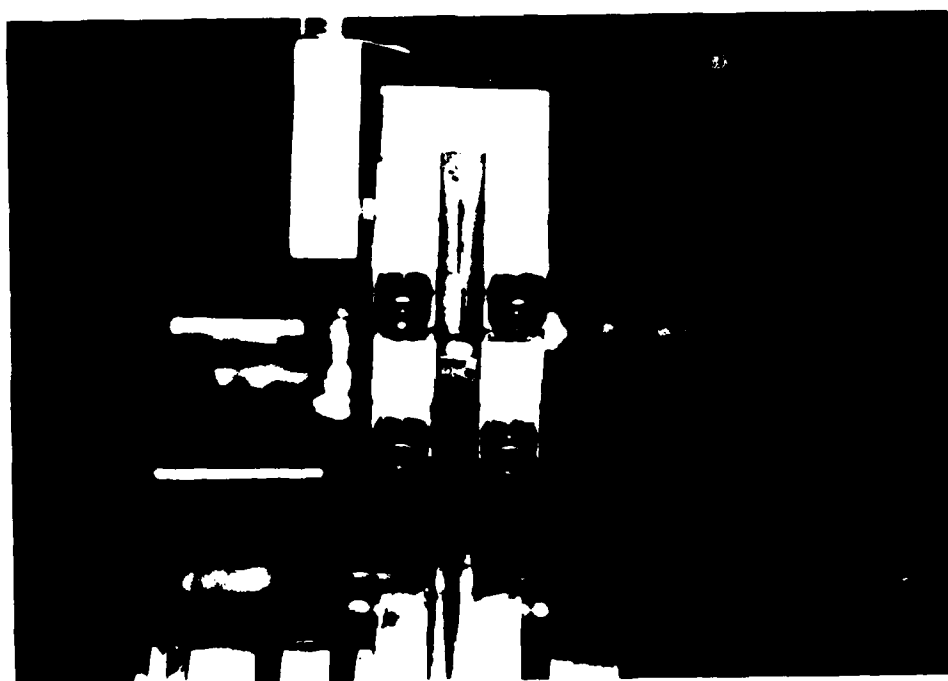
A. Plastic Well Arrangement

B. Specimen in Well

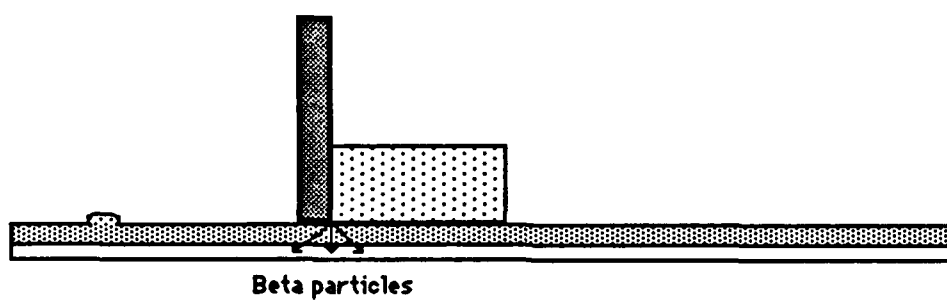


**Plate 12. Isomet Diamond Saw Sectioning Specimen**





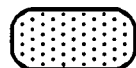
**Figure 2.** Specimen placement  
over radiographic film



EMULSION



FILM BASE

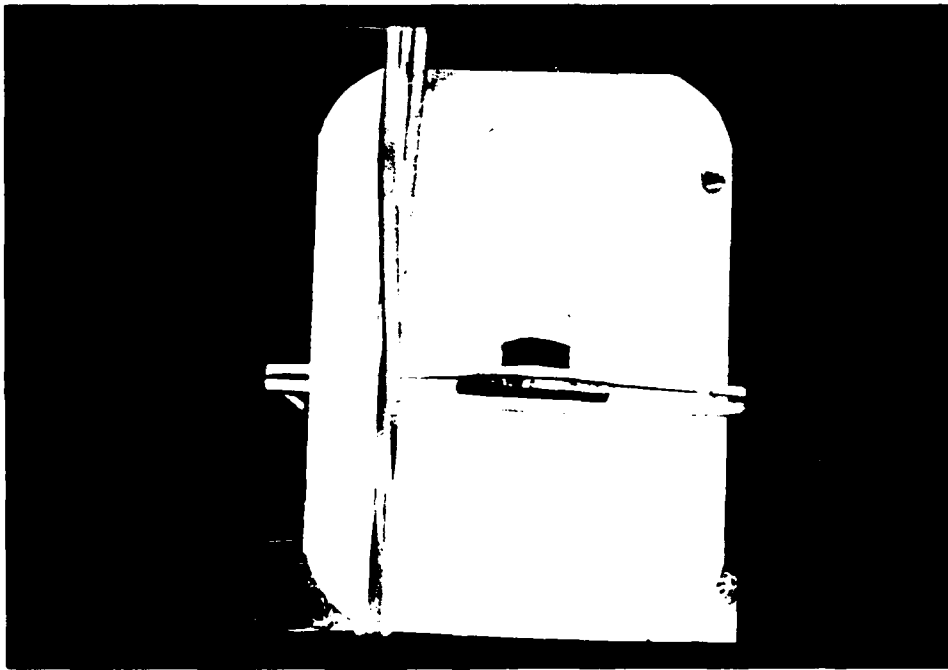


COMPOSITE RESIN



METAL

**Plate 13.** Placement of Sectioned Specimen over  
Radiographic Film



the composite resin. Three measurements were made and averaged to yield a mean.

### **I. Statistical Analysis**

The microleakage data of the specimens was analyzed by a two-way Analysis of Variance (ANOVA): retention systems (Beads, Silicoater, Lee and 4-META) and composite resins (Visio-Gem, Elcebond, Dentacolor). A statistical evaluation could not be made with 4-META and Lee specimens because of the limited number of specimens which did not fracture spontaneously. Therefore, a comparison only between Beads and the Silicoater system was made. A logarithmic transformation was used to correct the unequal variances between Beads and Silicoater groups. A Tukey's Multiple Comparison Test was made on all group means to identify significant differences.

## **V. RESULTS**

All of the 4-META specimens separated during thermocycling, demonstrating poor adhesion between the composite resin and metal. The location of failure could not be determined because 4-META is transparent.

Of the Lee specimens, more than 60% separated during sectioning with the Isomet diamond saw. All of these specimens exhibited an adhesive failure at the Lee opaquer-metal interface when evaluated through a stereomicroscope at 40X power.

Only data comparing the Silicoater and Beads was used for statistical analysis, due to the limited number of the specimens for the other systems. One specimen from the Beads/VG and one specimen from Silicoater/VG were lost during testing due to improper handling of the Isomet saw.

The raw microleakage data are listed in the Appendix; means and standard deviations are listed in Table 2 and 3 and Figure 3, the results of the two-way Analysis of Variance are shown in Table 4. Microleakage of Bead's had mean values of 4.09 mm, compared to Silicoater which had 0.04mm (Table 2). These values transformed into percentages yield a mean value close to 100% for beads, in contrast with Silicoater, which had mean values close to 0%. The Silicoater/Elcebond combination exhibited higher microleakage values with a mean of 0.11mm than Silicoater/Visiogem and Silicoater/Dentacolor that had 0.00mm (Table 3). These values transformed into percentages are 0.02% and 0% respectively (Table 5).

The results of the two-way ANOVA demonstrated a statistically significant difference ( $p < 0.001$ ) in levels of microleakage between Silicoater and Bead specimens (Table 4). A subsequent Tukey's Multiple Comparison test ranked microleakage: Silicoater/Visio-Gem less than Beads/Visio-Gem,

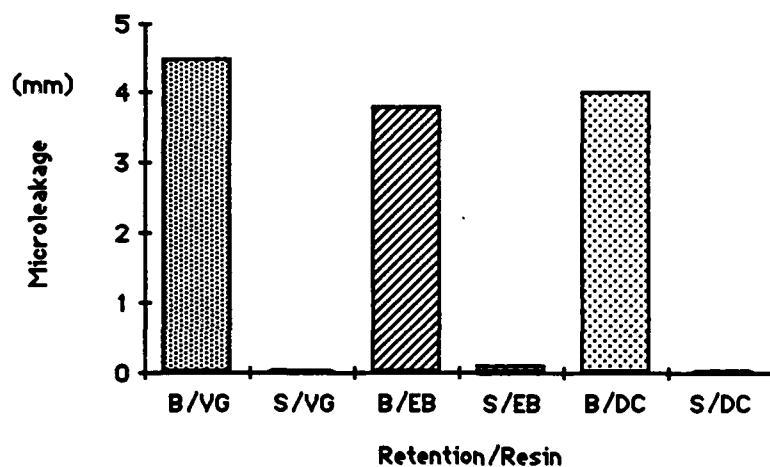
**Table 2.** Means and Standard Deviations of Microleakage of Retention and Resins

	<b>System</b>	<b>Mean(mm)</b>	<b>S.D.</b>	<b>N</b>
<b>Retention</b>	Beads	4.09	0.62	29
	Silicoater	0.04	0.13	29
<b>Resins</b>	Visiogem	2.24	2.31	18
	Elcebond	1.96	2.01	20
	Dentacolor	2.01	2.06	20

**Table 3.** Means and Standard Deviations of Microleakage of Retentive Systems

Retentive System	Mean(mm)	S.D.	N
Beads/VG	4.48	0.15	9
Beads/EB	3.81	0.94	10
Beads/DC	4.02	0.18	10
Silicoater/VG	0.00	0.00	9
Silicoater/EB	0.11	0.20	10
Silicoater/DC	0.00	0.00	10



**Figure 3. Cell Means Graph**

B/VG = Beads/Visio-Gem  
S/VG = Silicoater/Visio-Gem  
B/EB = Beads/Elcebond  
S/EB = Silicoater/Elcebond  
B/DC = Beads/Dentacolor  
S/DC = Silicoater/Dentacolor

**Table 4.** Summary Table for Two-way Analysis of Variance

Source	SS	df	MS	F	p
Main Effects	871.669	3	290.556	210.189	0.000
Reten	863.430	1	863.430	624.607	0.000
Resins	8.239	2	4.120	2.980	0.060
2-Way Interactions	11.537	2	5.768	4.173	0.021
Retention-Resins	11.537	2	5.768	4.173	0.021
Explained	880.089	5	176.018	127.332	0.000
Residual (Error)	71.883	52	1.382		
Total	951.972	57	16.701		

**Table 5. Summary Table**

RETENTIVE SYSTEMS	COMPOSITE RESINS		
	VISIO-GEM	ELCEBOND	DENTACOLOR
BEADS	100% leakage N = 9	93 % leakage N = 10	100% leakage N = 10
SILICOATER	0% leakage N = 9	2.48% leakage N = 10	0% leakage N = 10
LEE	80% broke at sectioning	70% broke at sectioning	60% broke at sectioning
4-META	100% broke at thermocycling	100% broke at thermocycling	100% broke at thermocycling

Silicoater/Elcebond less than Beads/Elcebond, Silicoater/Dentacolor less than Beads/Dentacolor ( $p < 0.001$ ) (Table 6). Silicoated specimens exhibited very little or no microleakage at the composite-metal interface when compared with microbeads.

The type of composite resin was not significant when used with Beads. However, the type of composite resin was significant when used with the Silicoater adhesive system ( $p = 0.021$ ) (2-way interaction in Table 4). The Tukey's Multiple Comparison Test demonstrated that the Silicoater/Elcebond combination exhibited higher microleakage values when compared to Silicoater/Visiogem and Silicoater/Dentacolor ( $P = 0.025$ ) (Table 6). However, mean values of microleakage of Silicoater/Elcebond are still very low when compared to Beads.

**Table 6. Tukey's Multiple Comparison Test Table**

<b>Retentive Systems</b>	<b>p</b>
Silicoater/VG - Beads/VG	0.000
Silicoater/EB - Beads/EB	0.000
Silicoater/DC - Beads/DC	0.000
Silicoater/VG - Beads/EB	0.000
Silicoater/VG - Beads/DC	0.000
Silicoater/EB - Beads/VG	0.000
Silicoater/EB - Beads/DC	0.000
Silicoater/DC - Beads/VG	0.000
Silicoater/DC - Beads/EB	0.000
Beads/VG - Beads/EB	0.999
Beads/EB - Beads/DC	0.999
Beads/VG - Beads/DC	0.999
Silicoater/VG - Silicoater/EB	0.025
Silicoater/EB - Silicoater/DC	0.025
Silicoater/VG - Silicoater/DC	1.000

## **VI. DISCUSSION**

The preparation of the specimens used for this investigation were accomplished by the same methods and materials used in clinical applications. The results of this investigation can be applied directly to the fabrication of dental prosthesis.

The fabrication of metal specimens, placement of adhesive and processing of the composite resin were accomplished following the manufacturers' instructions. A punch was designed and fabricated with specific dimensions so that a gasket could be made to provide a seal between the metal substructure and the teflon tube. The internal diameter was smaller than the tube. This prevented leakage of the composite resin before polymerization and provided a definite step in the composite resin, which was needed as a reference point for measurement with the traveling microscope.

The size of beads (0.4mm) in this investigation was based on what is available with the composite resin systems. Tanaka et al (1978) evaluated spherical particle size and retention, concluding that maximum retention and minimum dye penetration is achieved with 0.18mm beads. However, the particle size used in this investigation was adequate for retention purposes.

An opaque was not used in this investigation. Dentacolor (Kulzer) had the only composite resin (Bis-GMA) opaque available at the time of this investigation. Visio-Gem and Elcebond have opaques which are polymethylmethacrylate based. Polymethylmethacrylate does not bond to composite resin. It is used in systems where mechanical retention is being used exclusively. Visio-Gem has recently manufactured a composite resin (Bis-GMA) opaque and Elcebond (Teledyne-Hanau) is developing one also. An opaquer that is composed of a low viscosity composite resin would chemically bond to the composite resin resulting

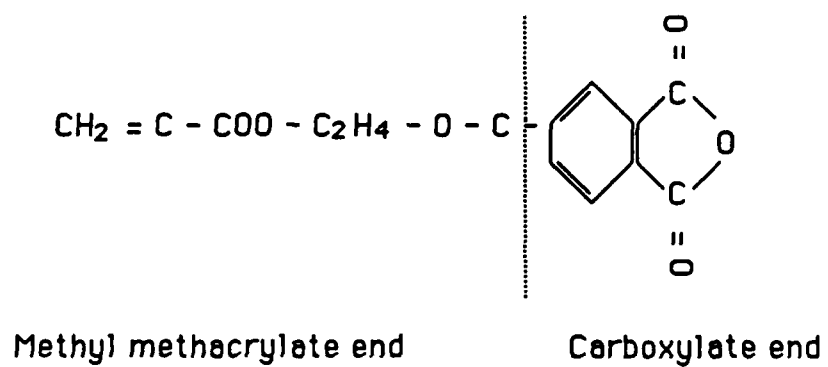
in a more retentive and stable restoration.

Visio-Gem is a composite resin which is packaged as a viscous liquid. Elcebond and Dentacolor, in contrast, are packaged as pastes. Application of Visio-Gem was made directly from the pre-packaged dispenser. However, Elcebond and Dentacolor were placed by extruding the material in the teflon tube and by lightly tapping the composite resin with the end of a spatula.

To provide clinical relevancy to this investigation, thermocycling was performed (Asmussen, 1974). Thermocycling has primarily been used for evaluation of direct composite resin materials on extracted teeth. This procedure simulates thermal stresses occurring in vivo over several years. Most investigators agree on temperatures between 4-60°C and less than 2,000 cycles with less than 1 min dwell (Crim and Mattingly, 1981; Hembree, 1983; Hembree and Taylor, 1984; Welsh and Hembree, 1985; Crim, Swartz and Phillips, 1985; Crim and García-Godoy, 1987).

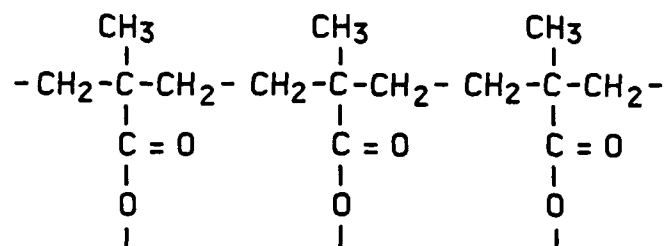
During the thermocycling procedure 100% of the 4-META specimens had the composite resin separate from the metal alloy. The location of the adhesive failure could not be determined because 4-META is colorless. 4-META is a bifunctional molecule (Figure 4) with a methacrylate group which has affinity to composite resin and a carboxylate group which bonds to metal (Figure 5). Metal carboxylates are not stable during thermocycling because these bonds hydrolize and eventually lose adhesiveness (Grieve, 1969; Going and Mitchem, 1975). The bond of 4-META has been reported as initially strong. Tensile bond values for noble metals averaged 20 MPa compared to 26 MPa in NiCr alloys. However, these values decreased gradually when stored in water (Wada, 1986). The bond strength appears to have decreased at a faster rate when the specimens were thermocycled, separating the composite resin from the metal. Air abrading with 50µm alumina powder and later depositing a tin layer electrolytically over gold

**Figure 4.** Molecular formula of 4-META

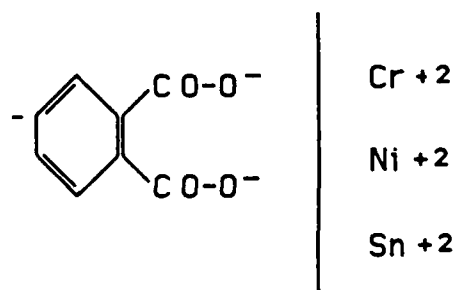




**Figure 5. Mechanism of adhesion of 4-META**



Methyl methacrylate polymerization



Metal Carboxylate bond

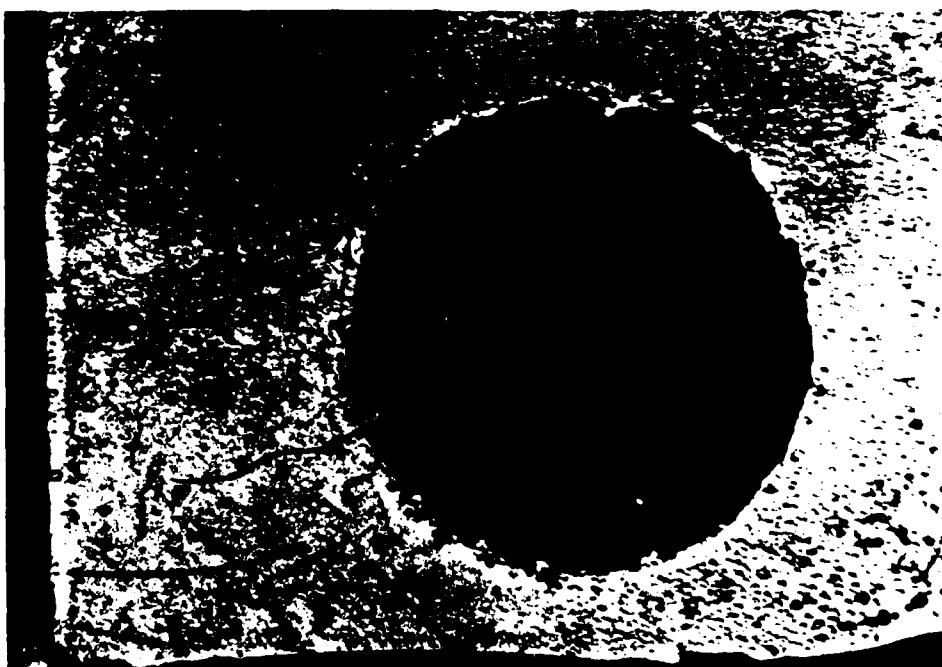
alloy surfaces has recently been reported as necessary to obtain reproducible results with adhesion promoters based on 4-META (Veen, 1985; Yamashita et al., 1986).

In more than 60% of the Lee specimens, the composite resin separated from the metal as soon as the blade of the Isomet saw made contact. A 40X power stereomicroscope was used to evaluate the interface. Failure was adhesive at the Lee Primer-metal interface (Plate 14). The bond of the Lee Primer-composite resin appears to be greater than the Lee Primer-metal. Some of the specimens that did not separate exhibited very little or no microleakage, which could lead to the conclusion that the bond is resistant to thermocycling but not resistant to mechanical forces. A study imbedding the specimens in acrylic resin prior to sectioning could clarify if the specimens exhibit microleakage after thermocycling.

Little is known of the chemical composition of Lee Metal Primer. The manufacturer states that it is an epoxy resin with a high molecular weight, which in its polymerized state is inert. The organic portion of the primer has been used in industry to line containers of food and beverages. The composition is: 22% epoxy resin and curing agent, 33% titanium dioxide plus other pigments, and 45% solvents (Lee Pharmaceuticals, 1985). Due to the limited information on its composition it can only be theorized based on the results of this investigation, that there are two possible mechanisms for the epoxy resin to adhere to the composite resin or metal. The adhesiveness of epoxy resins could probably be due to pendant secondary hydroxyl groups along the molecular chain, which are strongly absorbed on to oxide and hydroxyl surfaces (Potter, 1970). The other possibility is that after polymerization of epoxy resins there are no free-end radicals that could react with composite resin or metal. It is assumed that an additional group would have to be added to react with the composite resin. A

**Plate 14.** Representative of Lee Metal Primer after Thermocycling and Sectioning

- A. Adhesion of the Lee Metal Primer to the Composite Resin
- B. Adhesion Failure at the Metal - Lee Metal Primer Interface

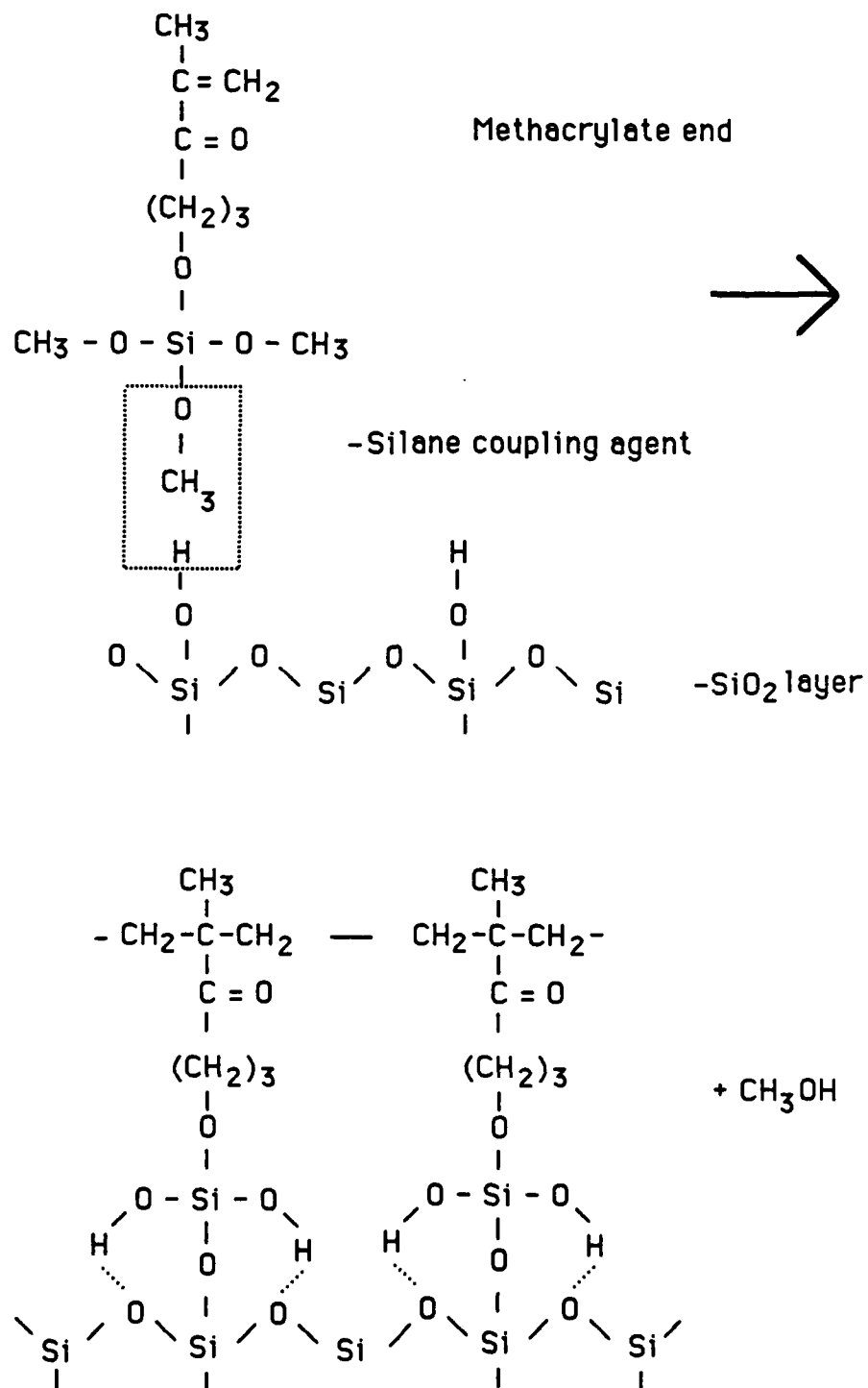


methacrylate could be added to the epoxy resin creating a pendant methacrylate group that could react with the composite. This could explain why the specimens bonded to the Lee Primer-composite resin interface. Failure at the Lee Primer-metal could be due to the limited amount of oxides of gold at the surface to provide adequate bonding with the pendant secondary hydroxyl groups and/or bonds not resistant to hydrolysis. The 50 $\mu$ m aluminum oxide abraded surface provides micromechanical retention to which epoxy resin can adhere. Epoxy resin adhesives are generally poor in peel strength. In addition, differences in coefficients of thermal expansion of the resins and metal (Potter, 1970) and thermocycling are factors that may have caused failure at the metal-primer interface.

In a preliminary report, Naegeli et al. (1985), evaluated shear bond strengths of Lee Primer and Silicoater specimens. Lee Primer exhibited adhesive failures at the metal-opaque interface (Visio-Gem and Dentacolor composite resins were used, with a gold containing alloy (Olympia)). The Silicoater specimens displayed a cohesive failure with a considerable amount of composite remaining at the metal-opaque interface. In another study, Hero et al (1987) reported adhesion of Visio-Gem to Ag-Pd alloys by means of Silicoating. A four point bend apparatus was used for evaluation of bond strength. Values were higher on Silicoated specimens in comparison to 0.6mm microbeads. This seems to correlate with the findings in this microleakage investigation. Silicoater appeared to have very little or no percolation at the composite-metal interface.

The Silicoater system consists of a pyrolytic deposition of silicon-oxide to a metal surface, to which a silane coupling agent is applied. The silane molecule is bifunctional, it reacts with the silicon-oxide surface by splitting off methanol and forming a Si-O-Si bridge. Pendant polymerizable groups of the silane polymerize with methacrylate groups of the composite resin (Figure 6). The net effect is a

Figure 6. Mechanism of adhesion of Silicoater



bond between metal and composite resin.

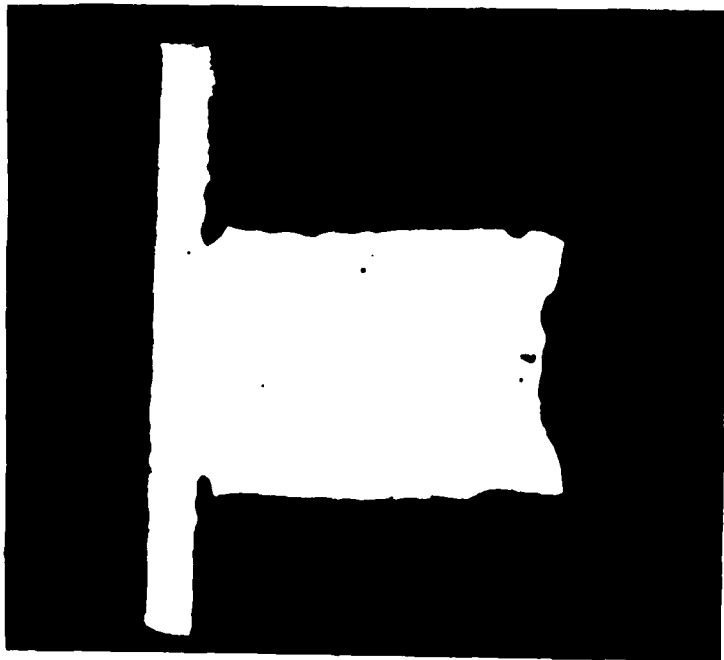
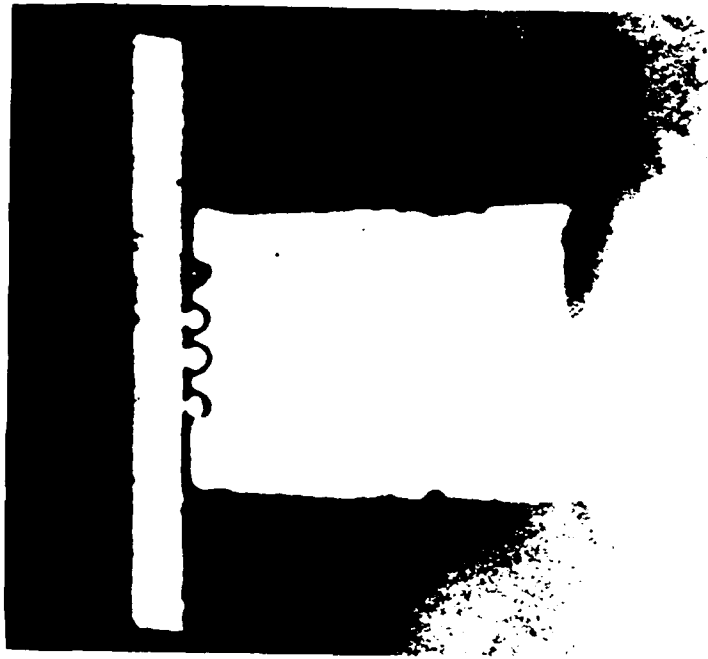
Methylene blue dye was used originally in this study as a marker. However, the results were not satisfactory. It did not provide adequate contrast and sharp demarcation between the metal and the composite resin.  $\text{Ca}^{45}$  was chosen as a marker after a study by Staffanou et al in 1985, which evaluated esthetic veneering materials like Pyroplast, Isosit and porcelain. A pilot study was made using the protocol of Staffanou et al (1985). However, no evidence of radioactivity at the interface was found. Another pilot study was necessary to determine the minimum amount of immersion time of the specimens in the radioisotope, and the optimum time required for autoradiography. Using two previous reports (Swartz and Phillips 1957, Phillips, 1961) as references, concentrations of 0.1mCi/ml and 0.2mCi/ml were used. This was 1000 times more than Staffanou's concentration (0.1 $\mu$ Ci/mm, sic). Immersion time was varied from 2 to 24 hours. Specimens were placed face down for 13 hours to 7 days on dental x-ray film for autoradiography. The pilot study indicated that an immersion time of 24 hours in a solution of  $\text{Ca}^{45}$  of 0.2mCi/ml, and an exposure time of 5 days for autoradiography provided optimum contrast for measurement at the interface without penetration of the radioisotope into the composite resin. The autoradiograph revealed a dramatic difference between Bead and Silicoater groups (Plate15). The composite resin-metal interfaces of the Beads specimens were permeable to the  $\text{Ca}^{45}$ . This was in contrast with the Silicoater specimens which were impermeable.

In previous microleakage investigations, measurement between composite resin and natural teeth was performed by assigning an arbitrary value ( 0, 1, 2, 3, 4, etc) to an anatomical (enamel, dentin, pulp) or cavity preparation landmark (dentinoenamel junction, axial wall, pulpal wall, etc). Dye or radioisotope penetration was measured by comparing the relative position of the marker to the established landmark. Examples of this are: 0 = no penetration, 1 = penetration

**Plate 15. Autoradiographs**

- A. Bead Specimen Representing Microleakage**
- B. Silicoated Specimen without Microleakage**





into 1/2 dentin width, 2 = penetration of total depth of cavity preparation and 3 = penetration of dye into pulp chamber (Fuchs et al, 1985) For methyl methacrylate resin, composite resin and porcelain veneering systems, evaluation of the interface has been divided into percentages or fractions. For example: 0 = no penetration, 1 = slight penetration up to 1/4 of the interface, 2 = penetration up to 1/2, 3 = penetration up to 3/4, 4 = complete penetration ( Staffanou et al, 1985). An ordinal scale of measurement with assigned values describing microleakage penetration is subject to error due to the variability of interpretation by the observer. Also, only discrete variables can be obtained, interfering with comparison of data with other studies and application of parametric statistics. This investigation demonstrates that direct measurement of the interface can be possible, in which parametric statistics can be applied.

The Silicoater system was the most effective bonding system of all adhesive systems studied. The silicon-oxide-carbon coating and silane coupling agent appear to be an effective substrate for bonding to composite resin.

## **VII. SUMMARY**

The bonding of three composite resin veneering systems, Visio-Gem, Elcebond and Dentacolor to a Type IV gold alloy with three chemical retention systems, 4-META, Lee Metal Primer, Silicoater has been investigated and compared to microbeads. Specimens were thermocycled between 6°C and 60°C and microleakage was measured utilizing a  $\text{Ca}^{45}$  marker. The following conclusions and recommendations can be made from this investigation:

1. Microleakage of Visio-Gem, Elcebond and Dentacolor when used with the Silicoater System were statistically significantly less than with microbeads on Type IV gold.
2. Microleakage was found to be statistically significantly higher with Elcebond than with the other composite resins, Visio-Gem and Dentacolor.
3. Inadequate bond was observed with 4-META. However, the weak interface could not be identified.
4. Adhesive bond failure was observed with Lee Metal Primer to the gold alloy.
5. Further research is needed to determine the long term effect of tin plating of noble metal alloys prior to placement of adhesives containing 4-META.

**APPENDIX**

### Raw Data for Test Specimens

Specimen#	Composite (mm)	Leakage (mm)	Leakage %
1	4.554	4.554	100
2	4.232	4.232	100
3	4.442	4.442	100
4	Broke (improper handling)		
5	4.543	4.543	100
6	4.381	4.381	100
7	4.650	4.650	100
8	4.701	4.701	100
9	4.382	4.382	100
10	4.407	4.407	100
11	4.403	4.403	100
12	4.474	1.438	32.1
13	4.305	4.305	100
14	3.911	3.911	100
15	4.266	4.266	100
16	4.295	4.295	100
17	3.715	3.715	100
18	3.046	3.046	100
19	4.400	4.400	100
20	4.339	4.339	100
21	4.039	4.039	100
22	4.123	4.123	100
23	3.781	3.781	100
24	3.979	3.979	100
25	3.800	3.800	100
26	3.896	3.896	100
27	4.040	4.040	100
28	3.921	3.921	100
29	4.296	4.296	100
30	4.278	4.278	100
31	4.542	0	0
32	Broke (improper handling)		
33	4.564	0	0
34	4.459	0	0
35	4.492	0	0
36	4.423	0	0
37	4.544	0	0
38	5.534	0	0
39	4.488	0	0
40	4.590	0	0

**Continuation of Raw Data for Test Specimens**

<b>Specimen#</b>	<b>Composite (mm)</b>	<b>Leakage (mm)</b>	<b>Leakage %</b>
41	4.272	0.521	12.20
42	2.814	0	0
43	4.456	0	0
44	3.785	0	0
45	4.580	0	0
46	4.632	0.442	9.54
47	4.378	0.136	3.10
48	4.195	0	0
49	3.840	0	0
50	4.154	0	0
51	4.059	0	0
52	4.434	0	0
53	4.384	0	0
54	4.197	0	0
55	4.269	0	0
56	4.101	0	0
57	4.378	0	0
58	4.250	0	0
59	4.320	0	0
60	4.222	0	0

# Raw Data on Specimens Not Used in Statistics

Specimen#	Composite (mm)	Leakage (mm)	Leakage %
61	Broke during sectioning		
62	Broke during sectioning		
63	Broke during sectioning		
64	Broke during sectioning		
65	3.979	0	0
66	Broke during thermocycling		
67	Broke during sectioning		
68	4.222	0	0
69	Broke during sectioning		
70	Broke during sectioning		
71	Broke during sectioning		
72	4.108	0.291	7.08
73	Broke during sectioning		
74	Broke during sectioning		
75	Broke during sectioning		
76	Broke during sectioning		
77	4.058	0	0
78	Broke during sectioning		
79	3.772	0	0
80	Broke during sectioning		
81	Broke during sectioning		
82	3.818	3.818	100
83	Broke during sectioning		
84	3.335	0	0
85	Broke during sectioning		
86	Broke during sectioning		
87	4.055	4.055	100
88	4.216	0.569	13.49
89	Broke during sectioning		
90	4.006	4.006	100

# Specimen Designation and % of Microleakage

Specimen#	Retention	Resins	% Leakage
1	Beads	VG	100
2	Beads	VG	100
3	Beads	VG	100
4	Beads	VG	Broke (improper handling)
5	Beads	VG	100
6	Beads	VG	100
7	Beads	VG	100
8	Beads	VG	100
9	Beads	VG	100
10	Beads	VG	100
11	Beads	EB	100
12	Beads	EB	32.1
13	Beads	EB	100
14	Beads	EB	100
15	Beads	EB	100
16	Beads	EB	100
17	Beads	EB	100
18	Beads	EB	100
19	Beads	EB	100
20	Beads	EB	100
21	Beads	DC	100
22	Beads	DC	100
23	Beads	DC	100
24	Beads	DC	100
25	Beads	DC	100
26	Beads	DC	100
27	Beads	DC	100
28	Beads	DC	100
29	Beads	DC	100
30	Beads	DC	100
31	Silicoat	VG	0
32	Silicoat	VG	Broke (improper handling)
33	Silicoat	VG	0
34	Silicoat	VG	0
35	Silicoat	VG	0
36	Silicoat	VG	0
37	Silicoat	VG	0
38	Silicoat	VG	0
39	Silicoat	VG	0
40	Silicoat	VG	0



**Continuation of Specimen Designation and % of Microleakage**

<b>Specimen#</b>	<b>Retention</b>	<b>Resins</b>	<b>% Leakage</b>
41	Silicoat	EB	12.20
42	Silicoat	EB	0
43	Silicoat	EB	0
44	Silicoat	EB	0
45	Silicoat	EB	0
46	Silicoat	EB	9.54
47	Silicoat	EB	3.10
48	Silicoat	EB	0
49	Silicoat	EB	0
50	Silicoat	EB	0
51	Silicoat	DC	0
52	Silicoat	DC	0
53	Silicoat	DC	0
54	Silicoat	DC	0
55	Silicoat	DC	0
56	Silicoat	DC	0
57	Silicoat	DC	0
58	Silicoat	DC	0
59	Silicoat	DC	0
60	Silicoat	DC	0

# **Specimens Not Used In Statistics**

## **Specimen Designation and % of Microleakage**

---

<b>Specimen#</b>	<b>Retention</b>	<b>Resins</b>	<b>% Leakage</b>
<hr/>			
61	Lee	VG	Broke during sectioning
62	Lee	VG	Broke during sectioning
63	Lee	VG	Broke during sectioning
64	Lee	VG	Broke during sectioning
65	Lee	VG	0
66	Lee	VG	Broke during thermocycling
67	Lee	VG	Broke during sectioning
68	Lee	VG	0
69	Lee	VG	Broke during sectioning
70	Lee	VG	Broke during sectioning
71	Lee	EB	Broke during sectioning
72	Lee	EB	7.08
73	Lee	EB	Broke during sectioning
74	Lee	EB	Broke during sectioning
75	Lee	EB	Broke during sectioning
76	Lee	EB	Broke during sectioning
77	Lee	EB	0
78	Lee	EB	Broke during sectioning
79	Lee	EB	0
80	Lee	EB	Broke during sectioning
81	Lee	DC	Broke during sectioning
82	Lee	DC	100
83	Lee	DC	Broke during sectioning
84	Lee	DC	0
85	Lee	DC	Broke during sectioning
86	Lee	DC	Broke during sectioning
87	Lee	DC	100
88	Lee	DC	13.493
89	Lee	DC	Broke during sectioning
90	Lee	DC	100

# Continuation of Specimens Not Used in Statistics

## Specimen Designation and % of Microleakage

Specimen#	Retention	Resins	% Leakage
91	4-META	VG	Broke during thermocycling
92	4-META	VG	Broke during thermocycling
93	4-META	VG	Broke during thermocycling
94	4-META	VG	Broke during thermocycling
95	4-META	VG	Broke during thermocycling
96	4-META	VG	Broke during thermocycling
97	4-META	VG	Broke during thermocycling
98	4-META	VG	Broke during thermocycling
99	4-META	VG	Broke during thermocycling
100	4-META	VG	Broke during thermocycling
101	4-META	EB	Broke during thermocycling
102	4-META	EB	Broke during thermocycling
103	4-META	EB	Broke during thermocycling
104	4-META	EB	Broke during thermocycling
105	4-META	EB	Broke during thermocycling
106	4-META	EB	Broke during thermocycling
107	4-META	EB	Broke during thermocycling
108	4-META	EB	Broke during thermocycling
109	4-META	EB	Broke during thermocycling
110	4-META	EB	Broke during thermocycling
111	4-META	DC	Broke during thermocycling
112	4-META	DC	Broke during thermocycling
113	4-META	DC	Broke during thermocycling
114	4-META	DC	Broke during thermocycling
115	4-META	DC	Broke during thermocycling
116	4-META	DC	Broke during thermocycling
117	4-META	DC	Broke during thermocycling
118	4-META	DC	Broke during thermocycling
119	4-META	DC	Broke during thermocycling
120	4-META	DC	Broke during thermocycling

### COMPUTATIONS FOR RADIOISOTOPE SOLUTION

A working solution was made to attain a concentration of 0.2mCi/ml. Verification of the concentration of the radioisotope solution was made by dilluting to 1/10 and counting duplicate 6 $\mu$ l aliquots in Liquid Scintillation solution and placing them through a Liquid Scintillation Spectrometer (Beckman model 1800).

Sample #1 = 259,460

Sample #2 = 268,420

Average of the two samples was 263,940 aprox 264,000 =  $2.64 \times 10^5$ dpm

6 $\mu$ l  $\xrightarrow{\text{gs}}$  60 $\mu$ l = 1/10 dillution ; 6 $\mu$ l  $\rightarrow$   $2.64 \times 10^5$ dpm

$2.64 \times 10^5$ dpm

X 10 dil. factor

$2.64 \times 10^6$  dpm/6  $\mu$ l ;  $\frac{1000 \mu\text{l/ml}}{6 \mu\text{l/aliquot}} = 166.7$  aliquot/ml

$2.64 \times 10^6$  dpm/6  $\mu$ l

X 166.67

$440 \times 10^6$  dpm/ml =  $0.44 \times 10^9$  dpm/ml

1mCi =  $2.2 \times 10^9$  dpm/mCi

$\frac{0.44 \times 10^9 \text{ dpm/ml}}{2.2 \times 10^9 \text{ dpm/mCi}} = 0.2\text{mCi/ml} = 0.12\text{mCi}/600\mu\text{l}$

**Batch Numbers of Materials Used**

Visio-Gem - N 240MD 082886

Elcebond - 50 3359

60 1469

51 2452

Dentacolor - 308023

507029

310124

401022

506021

Lee Metal Primer - 5303

4-META - (Cover-Up Kit) 70401, 55330

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### VITA

Luis James Blanco [REDACTED]  
[REDACTED]

He was raised in Puerto Rico and graduated from the University High School in 1967. He entered the University of Puerto Rico that same year where he majored in pre-medicine.

In the fall of 1971 he entered the University of Puerto Rico Dental School, in San Juan, Puerto Rico, and received a degree in Doctor of Dental Medicine in May, 1975. He was married to Adda Isabel Mascaró later that year. They have three children, [REDACTED]  
[REDACTED]

Upon graduation from dental school he accepted a partnership with his father , a prosthodontist and was in private practice, in Rio Piedras, Puerto Rico, until July, 1978. In addition to private practice, he was employed part-time by the City of San Juan Public Health Department.

In the summer of 1978, he resigned from his partnership in private practice and position with the City of San Juan Health Department and accepted a commission in the United States Air Force. He was assigned to Clark AFB, Philippines.

He was transferred to Dyess AFB, Texas, in October of 1980, where he served as Prosthetics Officer and Officer In Charge of the dental laboratory.

In June, 1984 he was readmitted to the University of Puerto Rico and completed his requirements for graduation, receiving the degree in Bachelor of Science in June 1985.

In July, 1985 he entered the University of Texas Health Science Center at San Antonio for graduate training in Prosthodontics, sponsored by the United States Air Force. In 1987 he was admitted as a candidate for the Master of Science Degree at the School of Biomedical Sciences. He received a certificate in Prosthodontics in June,

1988.

He has been assigned the position of Chief Prosthodontic Services at Robbins AFB, Georgia. He will assume this position in August 1988.